# Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

OCTOBER 1954

VOLUME 37 • NUMBER 10

# versatile...

# RHODAMINE BX CONC.
# RHODAMINE BXP CONC.

For pinks, for tinting whites, for brightening violets, RHODAMINE BX CONC. is useful in many grades of paper. Its unique shade and solubility characteristics make it applicable in many specialty papers.

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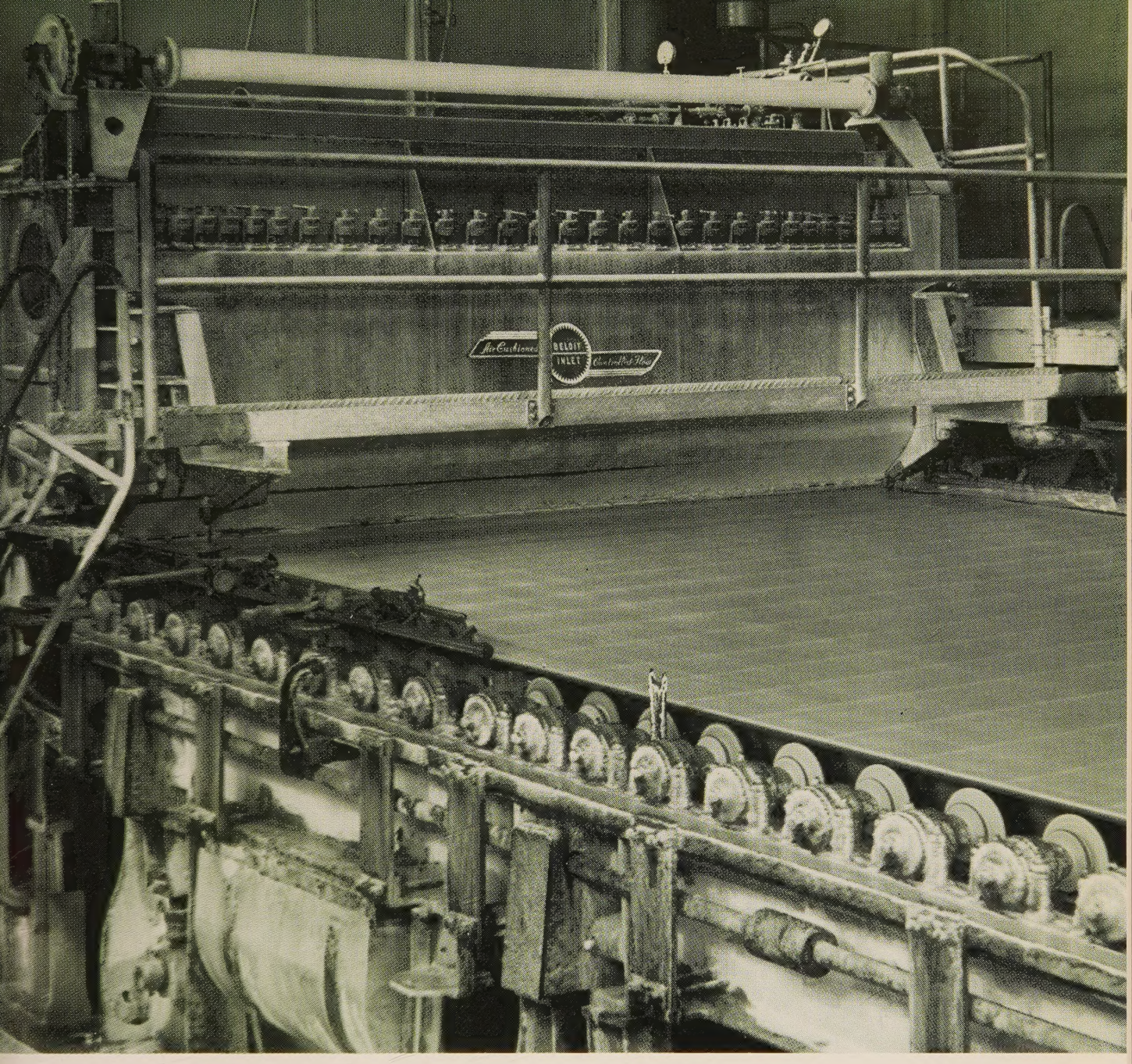


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**FIRST OF FOUR** patented Beloit Air-cushioned Inlets which are now in operation at Consolidated Water Power & Paper Company. Stock level inside is automatically held constant at all machine speeds. Self-cleaning rectifier rolls and controlled approach flow help to produce a level, high-quality sheet of uniform caliper.—*Beloit Iron Works, Beloit, Wisconsin.*

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*WHEN YOU BUY BELOIT... YOU BUY MORE THAN A MACHINE!*

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## ON THE GO IN THE MOTOR CITY

Smart retailers are quick to see the value of packages in color as "traveling advertisements" for their stores. A unique color is the best way for a store to put its distinctive personality into every package.

You'll find it profitable to help your retailer-customers achieve this prominent store identification. For, when you've helped your customer select a color that looks "tailor-made" for him, he'll want to use his color on wrapping paper, bags, boxes and gummed tape—every kind of packaging material you sell.

There are hundreds of shades you can choose from in adapting a distinctive package color for each store in town. Call on Du Pont for help in color selection and for technical assistance in selecting the right dye. For information, write to E. I. du Pont de Nemours & Co. (Inc.), Dyes and Chemicals Division, Wilmington 98, Delaware.

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*Du Pont dispersed organic pigments:*

"MONASTRAL"* Fast Blues, "MONASTRAL"* Fast Greens, "LITHOSOL"* Pigments

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*more use per dollar*

*with* **MANHATTAN**

*rubber covered rolls*

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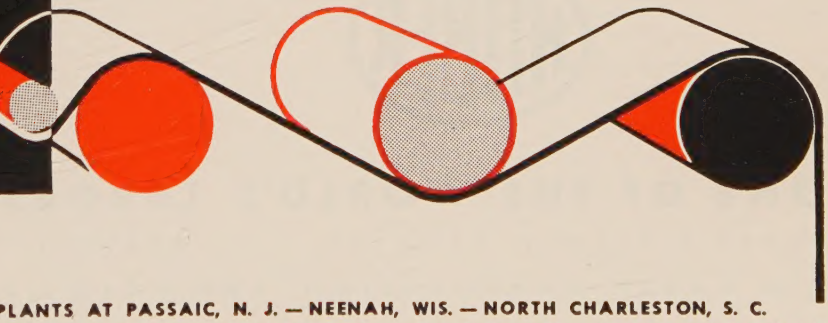

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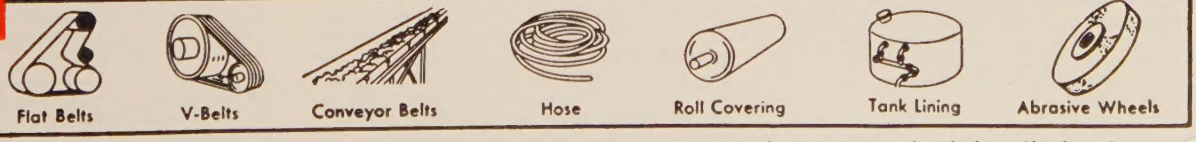


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RM 410

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79 Quality Checks Mean Finer Printing for You!
Quick death for bark! In huge rotating steel drums, logs are stripped of bark as they pound and rub together. Removal of all such undesirable elements helps make Kimberly-Clark papers whiter, cleaner, smoother. And for greater strength, exclusive new LongLac fibers are now included in all four coated papers.
3 TONS OF TRUFECT READY FOR SUPERCALENDERING—AND FURTHER QUALITY CONTROL CHECKS.
Makeup for a paper's "complexion"! As an aid in imparting the proper surface to all Kimberly-Clark papers, only soft, snow-white clay of finest texture is used in the coating formulation. And now, a finer balance of coating ingredients gives this paper the smoothest printing surface in Kimberly-Clark history.
How bright is bright? At Kimberly-Clark the brightness test tells—and as groundwood stock is dewatered, each sample taken must pass this test. It's just one of 79 checks on paper quality made from tree to press—79 reasons why Kimberly-Clark papers give you the press performance and reproduction of higher-priced paper.
Now you can make every impression a far better impression—without an increase in printing cost! For at Kimberly-Clark, the industry's most extensive quality control system assures premium quality press performance and reproduction—at the cost of ordinary paper!
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So, regardless of your paper requirements—for long runs or short runs, advertising pieces, magazines or house organs—look to Kimberly-Clark for printability at its best.
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CORPORATION
NEENAH, WISCONSIN
*T. M. REG. U. S. PAT. OFF.

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*As an aid in imparting the proper surface to all Kimberly-Clark papers, only soft, snow-white clay of finest texture is used in the coating formulation. And now, a finer balance of coating ingredients gives this paper the smoothest printing surface in Kimberly-Clark history."*

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**PARAGON**—produced in South Carolina, this low cost airfloated filler has a slightly larger particle size than Hi-White, its Georgia counterpart. Refined to the same close specifications with respect to abrasion and uniformity.

**WWF**—a Georgia water-washed filler of high, dependable brightness. Available in lump form for use where this type is preferred over the fluffier airfloated fillers. Also available pulverized.

**CWF**—a Georgia water-washed filler of same general type as WWF but with a slightly coarser particle size distribution which in some mills permits higher solids slurrying. Lump or pulverized.

**HYDRATEX**—a Georgia coating grade of medium particle size distribution and priced accordingly. Quality controlled throughout. Lump or pulverized.

**SPECIAL HYDRATEX**—a Georgia coating grade of fine particle size, with excellent covering power and flow characteristics ideally suited to certain coating methods. Lump or pulverized.

**X-43**—a fine particle Georgia coating clay of low viscosity and high brightness particularly well adapted to high solids coating. Lump or pulverized.

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| 2. Viscosity | 6. Adhesive Demand | 10. Soluble Salts |
| 3. Particle Size | 7. Gloss | 11. Abrasion |
| 4. pH | 8. Opacity | 12. Moisture |
| | | 13. Suspension |

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## TECHNICAL ARTICLES

The Flow Properties of Paper Pulp Stocks, *by Richard E. Durst and Lyle C. Jenness* ..... 417
Note on the Chemistry of the Cambial Zone of Black Spruce, *by Ernest Anderson, Louis E. Wise, and Evelyn K. Ratliff* ..... 422
Evaluation of the SFMC-TAPPI Drainage-Time Tester, *by C. E. Hrubesky and J. J. Perot, Jr.* ..... 425
Studies of Rosin Sizing by Means of Autoradiography and Electron Microscopy, *by Charles W. Hock* ..... 427
Sulphite Spent Liquor V, *by V. F. Felicetta, A. E. Markham, and Joseph L. McCarthy* ..... 431
Sizing Paper Under Neutral or Alkaline Conditions, *by W. F. Reynolds, L. A. Lundberg, and J. C. Barthel* ..... 436
An Investigation of Haze in Cellulose Acetates Made from Wood Pulps, *by Keith E. Bradway* ..... 440
Effects of Mechanical Preparation and pH on the Strength of Glass-Fiber Paper, *by Martin J. O'Leary, Robert B. Hobbs, J. K. Missimer, and James J. Erving* ..... 446
Arsenic Content of Pulp from Chemically Debarked Trees, *by Robert F. Smith and Karsten Mathisen* ..... 451
The Accelerated Biological Oxidation of Kraft Mill Effluents, *by Fred W. Bishop* ..... 453
The Beater Addition of Phenolic-Rubber Combination, Part I, *by R. Krajewski and V. Stannett* ..... 455
Studies in Continuous Alkaline Pulping, *by W. J. Nolan and G. R. Kulkarni* .. 460
A Study on the Effects of Instrumental Variables on 75° Specular Gloss, *by L. W. Zabel and W. A. Wink* ..... 474
Erratum—The Effect of Different Pulps on the Biological Activity of Phenylmercuric Acetate, *by B. F. Shema and J. H. Conkey* ..... 480

## DEPARTMENTS

Ammonia-Base Sulphite Pulping, *by W. Q. Hull, B. C. Smith, J. H. Hull and W. F. Holzer.* 14 A
Progress in Alkaline Pulping—1953, *by J. E. Stone* ..... 70 A
Association News and Events ..... 103 A
Personal Mention ..... 108 A
Obituary ..... 145 A
Divisions and Committees ..... 146 A
Letters to the Editor ..... 151 A
TAPPI Standards
Tensile Strength of Paraffin Wax—TAPPI Tentative Standard T 644 m-54 ..... 154 A
Analysis of Clay—TAPPI Tentative Standard T 645 m-54 ..... 157 A
Brightness of Clay—TAPPI Tentative Standard T 646 m-54 ..... 159 A
Gross Particles in Coating Clay—TAPPI Suggested Method T 647 sm-54 ..... 160 A
Viscosity of Coating-Clay Slurry—TAPPI Suggested Method T 648 sm-54 ..... 161 A
Local Section Activities
The Application of Polyethylene and Polyethylene-Wax Blends by Extrusion and Roller Coating Methods, *by G. L. Booth* ..... 163 A
Modification of Plastic Films for Specific Packaging Needs, *by J. B. Sisson* ..... 170 A
Foam Measurement, *by H. T. Holland* ..... 172 A
Applications of the Hurletron Automatic Caliper and Basis Weight Control to Paper and Paperboard Machines, *by C. M. Noble* ..... 173 A
Developments in Flotation Deinking of Waste Paper, *by J. W. Jelks* ..... 176 A
Petroleum Waxes and Additives for Paper, *by W. S. Kumblad* ..... 180 A
Employment Service ..... 182 A
Shibley Award Papers
The "80-Grit-Touch-Up" Method for Maintaining Calibration of the Valley Laboratory Beater, *by F. Dinger* ..... 183 A
Methods and Results of a Kraft Mill Sodium Balance, *by M. L. Custis* ..... 187 A
Possible Improvements in the Routine Analysis of Ordnance Pulps, *by J. F. Farrington* 190 A
Variables Affecting Brightness of Unbleached Sulphite Pulp, *by D. L. Mosher* ..... 194 A
United States Patents on Papermaking—Second Quarter, 1954, *compiled by W. B. Weber* 197 A
Recent Books ..... 204 A
Calendar of TAPPI Meetings ..... 205 A

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Conveyor belt and storage bins at loading docks

# Crude Sulphur

**for Industrial Use**

*from the properties of*

## Texas Gulf Sulphur Co.

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*Producing Units*

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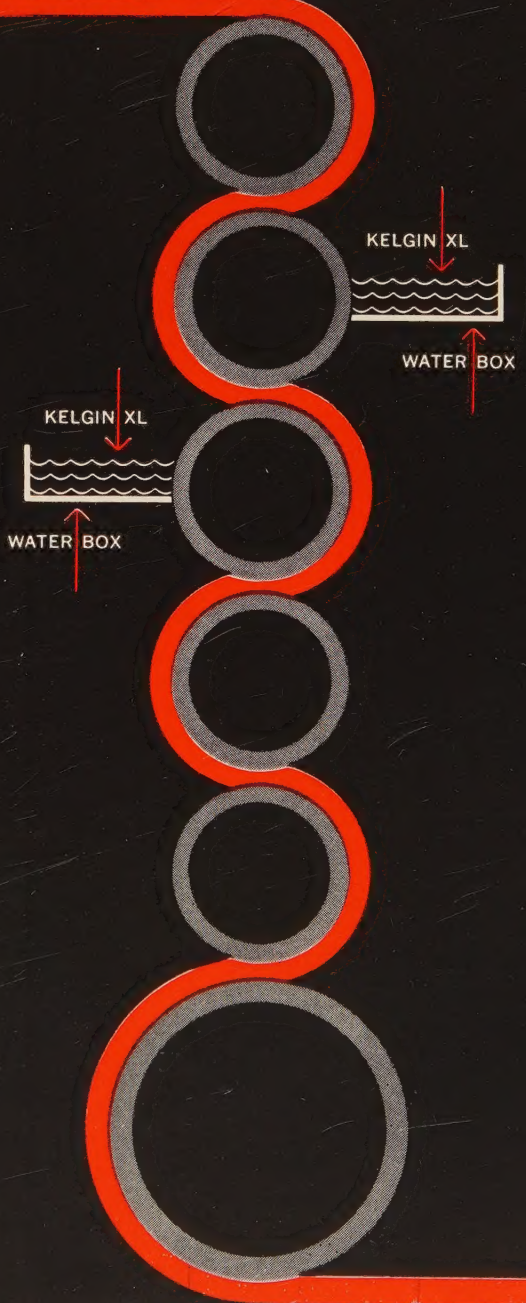
keep 'em rolling with

# KELGIN XL

non-tacky surface size




Because Kelgin XL size is non-tacky, it virtually eliminates calender wrapping. This hydrophilic colloid has excellent filming properties, produces improved smoothness, more uniform printing surface, and increased oil and varnish resistance without adverse effect on other paper qualities.

It is ordinarily applied from calender water boxes as a 1% to 3.5% solution with concentration used depending upon type and degree of finish desired.

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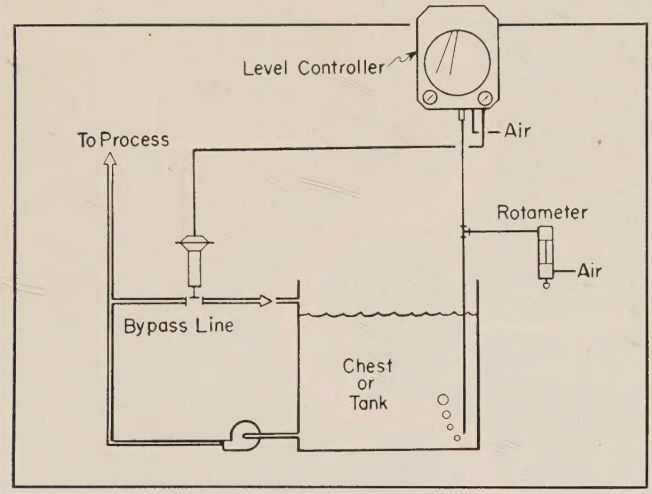
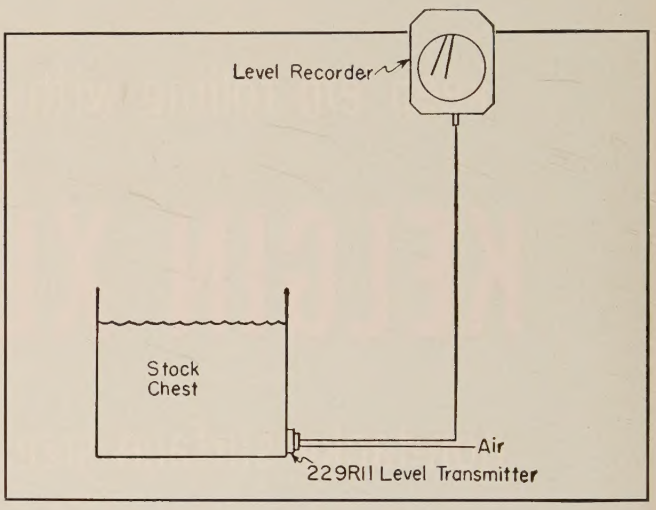
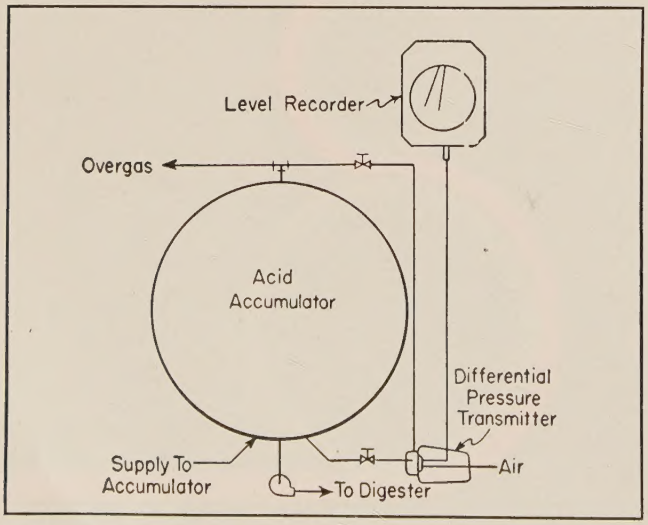



**Air Bubbler Method**—the most common method of measuring level in open tanks or chests. Utilizes back pressure caused by the hydrostatic head of liquid against an air-filled pipe in the tank, to operate a pressure instrument. Ideal for *stock chests, filters, sewer levels, white washer chests, etc.*




**Diaphragm Transmitters.** Recommended for use where a smooth surface or a sanitary or corrosion-resistant surface is required, or where no obstructions in tanks are permissible. Typical applications: *head boxes, high consistency stock tanks, liquor and chemical storage tanks, etc.*




**Differential Pressure Transmitters.** A mercuryless, force-balance transmitter, operating on a 3-15 lbs. output air pressure. Simplified piping; no seal pots; self-draining or venting. Applicable to any pressure vessel, e.g., *acid accumulators, washer seal boxes.* Also for *digester level.*

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*Instruments for indicating, recording and controlling temperature, pressure, flow, liquid level, speed, density, load and humidity.*

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# Johns-Manville CHEMPAC® PACKING withstands active acids and alkalis

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Chempac is outstanding in service against most acids and alkalis at temperatures to 500°F. It is especially recommended for use in pumps handling calcium bisulfite cooking liquor, sulfurous acid and relief gases . . . on relief valves on sulfite digesters . . . on valve stems exposed to chlorine, and for other equipment in service against corrosive liquids.

Chempac Packing is available in coil, spiral and ring form for rod, plunger and valve stem applications. Johns-Manville also manufactures Chempac Gaskets, made of plies of asbestos cloth treated with Teflon.

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*Trade mark for DuPont Tetrafluoroethylene resin

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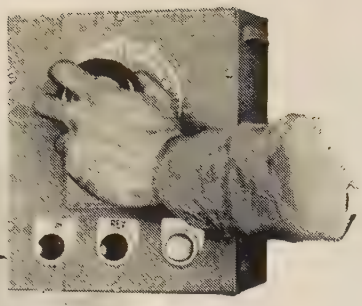
Operator's
Control Station



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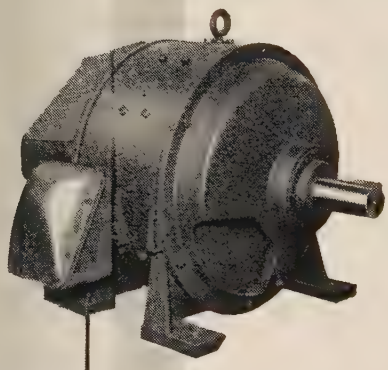
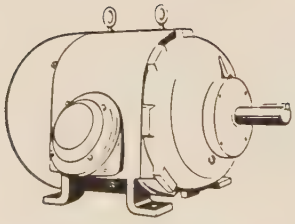
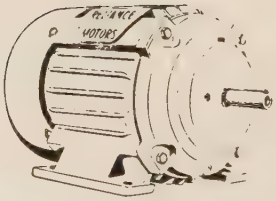
# THESE Tools of Automation BOOST

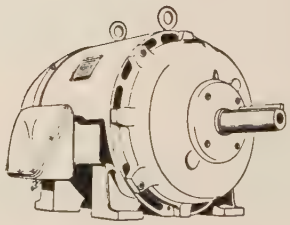
Control Unit

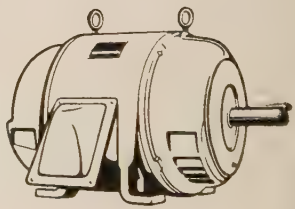


Adjustable-Speed
Drive Motor







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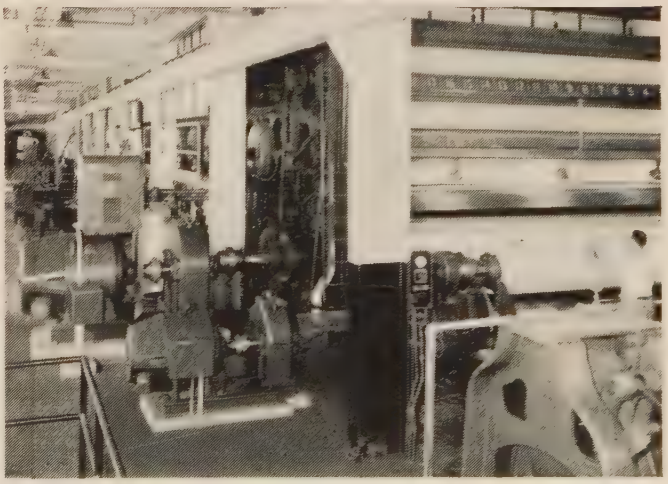

*On a high-speed newsprint machine, the Reliance V★S Drive system provides unsurpassed accuracy and dependability, maintaining precise speed relationships and correct draw adjustments between all sections.*

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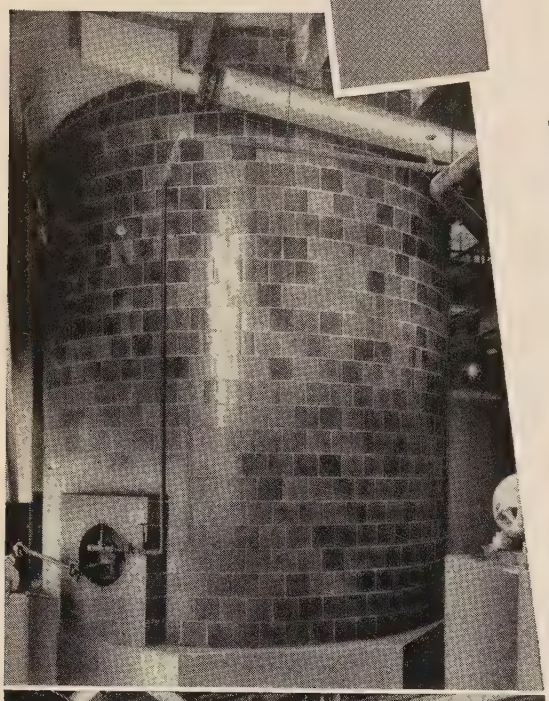

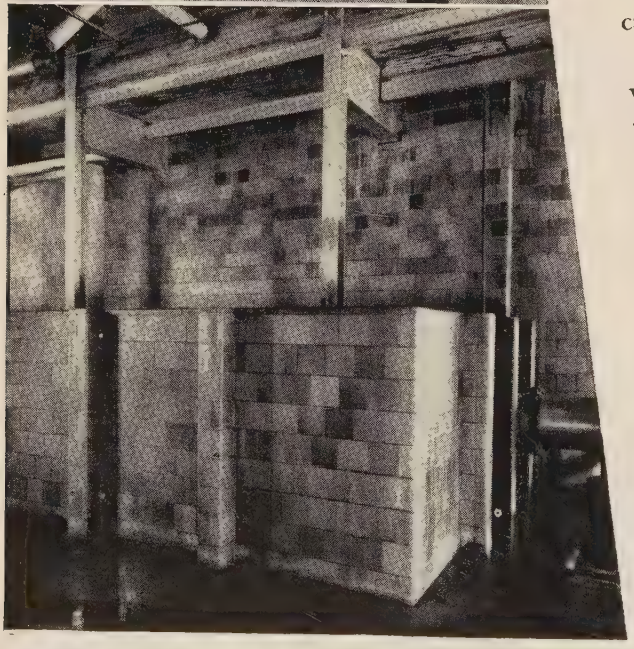

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# Ammonia-Base Sulphite Pulping

WILLIAM Q. HULL, BEVERLY C. SMITH, JAMES H. HULL, and WALTER F. HOLZER

EARLIER reports in this series described the romantic, centuries-old, history of pulp and papermaking. Two described kraft pulp production and kraft papermaking; a third portrayed comparatively new magnesia-base sulphite pulping (*7*, *14*, *15*). This article presents the ammonia-base pulping process, which is currently receiving more attention than any other in the field of introducing soluble base to normal sulphite pulping. Historical comment on processes not employing ammonia will not be repeated; too, most of the detailed description will be on unit operations and processes that are dissimilar to those of plants previously pictured in the staff-industry collaborative series.

In brief review, there are three principal processes for production of chemical wood pulp—soda, sulphite, and kraft (or sulphate). The order of production by the three processes is shown in statistics for 1953 (*17*) and in Table I.

| | *Thousand tons* |
|---|---|
| Soda | 428 |
| Sulphite | 2321 |
| Kraft | 9435 |
| Other chemical | 677 |
| Nonchemical and semichemical | 4664 |

The three chemical processes are all concerned with methods for removing the lignin, which binds the cellulose fibers in wood, and retaining the fibers with a minimum of degradation. Typically in the soda process, a 12.5% solution of sodium hydroxide and sodium carbonate in approximate ratio of 85:15 is used as a cooking liquor to dissolve noncellulosic materials from the wood; sodium hydroxide is responsible for the dissolving action. The kraft or sulphate process employs a mixture of sodium hydroxide and sodium sulphide. The sulphite process achieves the same results with an acid cooking liquor. In the long-used calcium bisulphite process, the cooking liquor composition is 7% by weight sulphur dioxide of which 4.5% is combined as sulphurous acid and 2.5% as calcium bisulphite (*16*). In magnesia-base sulphite pulping, magnesium oxide is substituted for limestone, and the resulting cooking liquor consists of magnesium bisulphite in an excess of sulphur dioxide.

New processes for pulpmaking have been sparked by problems of disposal of the spent cooking liquor and soluble components of wood as much as by the desirability of changes in the characteristics of the end product. The cellulose content of various woods in the United States ranges from 51 to 65% (*11*); this is the primary constituent of the pulp product. It is seen, therefore, that approximately half the wood entering a pulp mill is dissolved by the cooking liquor. In the sulphite process there is approximately 2000 gal. of spent liquor (12% solids) for each ton of pulp produced. In the standard calcium-base process, the solids consist of lignosulphonic acid (65%), reducing sugars (20%), sugar–sulphur dioxide derivatives (8.4%), and calcium (6.7%).

It is the disposal of huge volumes of spent liquor and its contained organic matter (estimated to be 4,900,000 tons yearly) that has been one of the major problems of the pulp industry. In earlier days, unrestricted discharge into streams was practiced; however, inasmuch as some of the sugars and organic acids become consumers of oxygen, the dissolved oxygen became depleted. In the past two decades, disposition of the liquor by other means has been investigated as a result of concentrated industrial and legal efforts toward stream improvement.

Many of the early efforts were in the direction of reducing the biological oxygen demand of the waste liquor making it suitable for stream discharge. Trickling filters, reaeration, and chemical alteration of the carbohydrate portion of the liquor were investigated. All added considerably to the cost of pulpmaking and none recovered the carbohydrate portion of the spent liquor. Others have converted the organic matter into useful compounds, including alcohol, yeast, vanillin, oxalic acid, tanning material, road binders, special cements, cement accelerators, and plastics. Such efforts have ranged from failure to commercial production. Production of food yeast from sulphite liquor

Compared with calcium-base pulping ammonia pulping has

Many advantages

- ...less sulphur per ton required
- ...decrease in cooking time
- ...less steam per ton
- ...less maintenance on digesters and pumps
- ...reduction in cost of handling and storing ammonia as compared with dolomite or lime rock
- ...more uniform acid making
- ...spent liquor not burned can be spray-dried and converted to salable by-products
- ...significant increase in physical strength and viscosity
- ...for paper, superior quality and uniformity

Few disadvantages

- ...slime growth in knotters to deckers system. Controlled by use of organomercurial slimicides
- ...redder pulp with western hemlock, but requires no additional chlorine for bleaching

Decrease in sulphur is attributed to decreased combined sulphur dioxide (from 1.30 to 0.85%) with no sacrifice in pulp properties except unbleached color. The faster cook cycle is due to quicker and more uniform penetration of chips by bisulphite cooking liquor and to the decreased combined sulphur dioxide below levels possible with calcium.

WILLIAM Q. HULL, Associate Editor, *Industrial and Engineering Chemistry*, BEVERLY C. SMITH, Crown Zellerbach Corp., Lebanon, Ore., JAMES H. HULL, Crown Zellerbach Corp., Camas, Wash., and WALTER F. HOLZER, Crown Zellerbach Corp., San Francisco, Calif.

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**Table I. U. S. Wood Pulp Production (17)**

(Thousands of short tons)

| | Dissolving and special alpha | Sulphite paper grades | | Sulphate paper grades | | | Soda | Total chemical | Groundwood | Defibrated, other non-chemical | Semi-chemical | Semi-screenings | Total, all grades |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Bleached | Unbleached | Bleached | Semi-bleached | Unbleached | | | | | | | |
| 1937 | 354 | 995 | 792 | 215 | | 1924 | 508 | 4,787 | 1601 | | 133 | 53 | 6,573 |
| 1938 | 228 | 776 | 602 | 321 | | 2123 | 395 | 4,445 | 1333 | | 119 | 37 | 5,934 |
| 1939 | 193 | 1024 | 729 | 424 | | 2538 | 442 | 5,351 | 1445 | | 152 | 46 | 6,993 |
| 1940 | 327 | 1285[a] | 996 | 453 | 131 | 3163 | 532 | 6,888 | 1633 | 210 | 165[a] | 64[a] | 8,960 |
| 1941 | 262 | 1442[a] | 1216 | 659 | 164 | 3704 | 480 | 7,925 | 1886 | 267 | 200[a] | 97[a] | 10,375 |
| 1942 | 404 | 1313[a] | 1213 | 653 | 159 | 3927 | 462 | 8,131 | 1870 | 406 | 210[a] | 167[a] | 10,783 |
| 1943 | 390 | 1163[a] | 883 | 602 | 147 | 3487 | 419 | 7,092 | 1767 | 495 | 231[a] | 96[a] | 9,680 |
| 1944 | 447 | 1076[a] | 863 | 665 | 144 | 3740 | 413 | 7,348 | 1769 | 532 | 285[a] | 175[a] | 10,108 |
| 1945 | 383 | 1161[a] | 816 | 705 | 149 | 3618 | 430 | 7,261 | 1827 | 560 | 295[a] | 224[a] | 10,167 |
| 1946 | 317 | 1376 | 784 | 750 | 152 | 3686 | 476 | 7,541 | 1951 | 616 | 320[a] | 178[a] | 10,607 |
| 1947 | 407 | 1495 | 894 | 909 | 193 | 4255 | 492 | 8,644 | 2050 | 693 | 444 | 114 | 11,946 |
| 1948 | 420 | 1490 | 902 | 1040 | 225 | 4749 | 510 | 9,335 | 2175 | 745 | 478 | 140 | 12,872 |
| 1949 | 374 | 1455 | 707 | 1118 | 264 | 4595 | 492 | 9,006 | 1960 | 604 | 506 | 131 | 12,207 |
| 1950 | 479 | 1629 | 741 | 1433 | 360 | 5709 | 522 | 10,872 | 2216 | 935 | 686 | 140 | 14,849 |
| 1951 | 616 | 1761 | 765 | 1900 | 294 | 6379 | 446 | 12,160 | 2474 | 945 | 797 | 150 | 16,524 |
| 1952 | 706 | 1692 | 673 | 2083 | 275 | 6211 | 425 | 12,065 | 2380 | 1059 | 829 | 140 | 16,473 |
| 1953 | 677 | 1767 | 594 | 2379 | 303 | 6753 | 428 | 12,861 | 2379 | 1116 | 1029 | 141 | 17,525 |

[a] Estd. from U. S. Bureau of Census combined data.

as practiced in the plant of Lake States Yeast Corp., Rhinelander, Wis., was described earlier in this series (*9*).

Second only to the problem of spent liquor disposal is consideration of the heat value of the dry organic solids in the spent liquor and recovery of cooking chemicals. The heat content ranges from 7000 to 8000 B.t.u. per lb. Many processes have been investigated for evaporating the calcium-base liquor and using the concentrated liquor as fuel. The concentrated liquor can satisfactorily be used under proper conditions for generation of steam.

However, inasmuch as the calcium and the sulphur combine during the burning operation to form calcium sulphate, from which the cooking compounds cannot be recovered practically, the processes have not been particularly attractive. Sodium has been substituted for calcium as a pulping base and is used in a limited number of mills. However, the recovery of the base as usable chemicals for the sulphite pulping process is complicated and costly.

Substitution of magnesium for calcium has been extensively investigated. In this process, magnesium is recovered in the form of magnesium oxide and is suitable for reuse as such. A history of the process and its commercial application in the 275-ton-per-day plant of Weyerhaeuser Timber Co. at Longview, Wash. (the only commercial magnesium oxide plant until recently) were related in this series (*7*). This plant converted to magnesia-base pulping following satisfactory pilot plant operation beginning in 1940. Overall operation and recovery have been satisfactory, and the system offers a definite method of stream improvement. Magnesium consumption is approximately 50 lb. per ton of pulp produced; sulphur consumption is 85 lb. per ton of pulp. With sufficient evaporator capacity, both these figures would be expected to be somewhat lower. They compare, nevertheless, with a consumption of approximately 270 lb. of sulphur and 350 lb. of lime rock for the conventional calcium-base pulping. A second magnesium-base plant has just begun operation at Ketchikan, Alaska. Owned by Ketchikan Pulp Co. (a combine between American Viscose Co. and Puget Sound Pulp and Timber Co.), the new plant, built at a cost of approximately $52,500,000, produces 350 tons of dissolving pulp daily. The magnesium oxide recovery system is probably more adequate, and it is expected that chemical consumption figures will be materially better than those experienced at the only prior mill using the process.

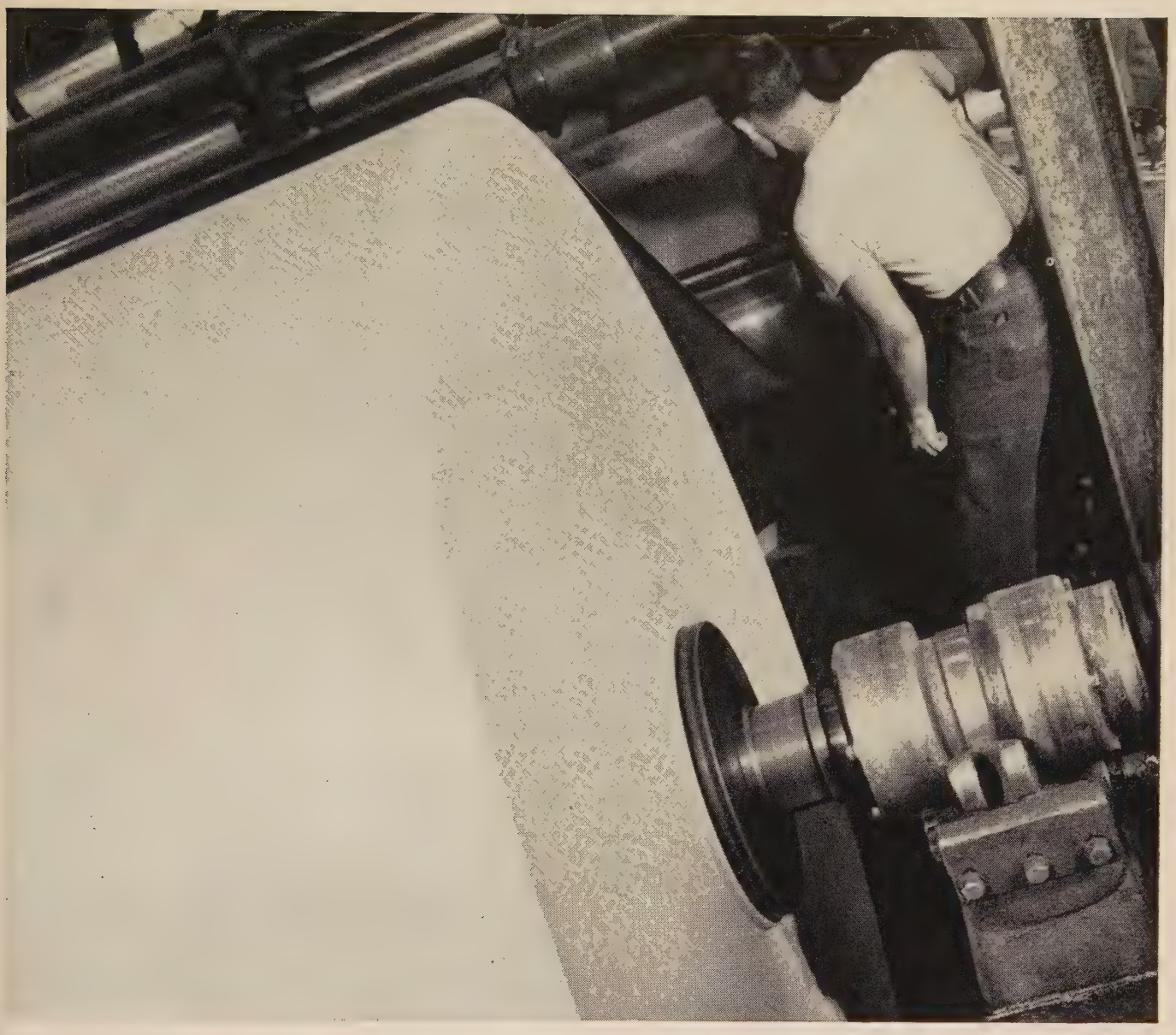
Magnesia-base pulping has several advantages: stream pollution is avoided; cooking chemicals can, in large measure, be recovered; and energy requirements of a mill can be supplied through the burning of the organic solids in the waste liquor. Yeast or alcohol can also be produced by running the partially concentrated liquor through an alcohol plant and returning the still slop to the recovery system.

A single, important disadvantage, however, is extremely high initial cost or cost of conversion. For this reason in existing mills the substitution of ammonia for conventional calcium is considered by many to be a more practical solution. However, any new sulphite mill will undoubtedly analyze the two processes thoroughly before deciding which it will use. This is particularly important because of the greatly increased production of ammonia during and since World War II.

With the recent and current interest that has been shown in the substitution of ammonia for calcium in the conventional sulphite process for making pulp, it would be reasonable to assume that the modified process is a new development. However, experimentally and commercially, the process has been known for many years. A patent was issued to Braun in Germany in 1902 covering an early version of ammonia pulping. As we know it today, the process was patented in England in 1923, Germany and France in 1924, and in the United States in 1925. The American patent (*3*) describes the system as:

> A sulphurous acid process for resolving wood substance, which process consists of heating the wood substance with an aqueous sulphurous acid solution to which has been added a calculated small amount of ammonia.

The first company to use the process commercially was Norsk Hydro-Eletstrisk Kvaelsofaktieselskab, a large Norwegian ammonia manufacturer, in its mill in Toten, Norway, in 1936. Reported advantages of



# "VIRGINIA" ZINC HYDRO

## bleaches groundwood pulp for about one-half the cost— saves the paper industry thousands yearly

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ammonia pulping as contrasted with ordinary calcium sulphite pulping at the time of conversion of this plant were a reduced cooking time at a lower temperature, a higher yield of cellulose per unit of wood, and a cellulose low in ash (*8*). The plant has used the process continuously since that time.

In the United States, ammonia-base pulping was pioneered commercially by Rayonier, Inc., whose plant at Shelton, Wash., was converted from calcium pulping in 1945. The plant has a capacity of 300 tons per day. Eastern Corp. followed with conversion of two plants in Maine in 1947 and 1948. The Lebanon mill of Crown Zellerbach, subject of this article, began in 1949. Wausau paper mills became the first midwestern mill to change over to ammonia cooking in 1950. Rayonier's second plant to convert, located at Hoquiam, Wash., produces 400 tons per day which is the largest ammonia-base production at any one plant.

The National Production Authority, U. S. Department of Commerce, surveyed American pulp mills in 1953 to determine the status of ammonia pulping. The study revealed the following information:

| | |
|---|---|
| Number mills surveyed | 62 |
| Number returns | 62 |
| Number mills using ammonia process | 10 |
| Number anticipating use in next 3 years | 9 |

| | *Annual pulp capacity, tons* | *Estimated annual ammonia consumption, tons* |
|---|---|---|
| 62 mills | 3,064,000 | ... |
| 10 mills now using | 505,000 | 26,250 |
| 9 mills probable | 338,000 | 18,050 |

The United States and Canadian mills actually using ammonia pulping at this time are shown in Table II (*10*).

Ammonia-base pulping, basically, is identical chemically with calcium pulping except that the ammonium ion is substituted for the calcium ion in the bisulphite cooking liquor. There are several advantages, however, over other modified sulphite processes. An important one is that the change-over is relatively simple and requires little modification of existing equipment. Because ammonia-base liquors do not have the scaling properties of calcium-base liquors, they are not adequately retained by linings used for calcium-base plants. These linings, however, can be replaced after conversion, as necessary in the regular maintenance program.

Principal additions to the plant are the storage and handling facilities for ammonia. The spent liquor can be burned satisfactorily, and stream pollution is eliminated. In addition, at least partial recovery of cooking chemicals is possible. A favorable consideration is possible utilization of some woods, such as jack pine, maple, beech, and others, normally difficult to pulp, as result of a better penetration rate and more favorable side reactions in ammonia pulping. (The mechanisms of chemical side reactions are not completely understood. This much seems certain, though perhaps oversimplified: products of side reactions due to chemicals in the wood together with ammonia salts are soluble whereas similar calcium products are not. Removal of the calcium base from the reaction by precipitation very seriously alters the course of the main reaction.)

Future conversion to ammonia pulping represents a sizable market for ammonia, and ammonia producers have cooperated closely with pulp mills in problems accompanying a change from calcium-base pulping. Nitrogen Division, Allied Chemical Dye Corp., has actively participated in the conversion of most of the mills other than Rayonier's, and Allied's observations of the process in a number of conversions have been published (*5*).

**Table II. North American Mills Using Ammonia-Base Pulping (*10*)**

| *Company* | *Location* | *Production, tons pulp/day* | *Date of conversion* |
|---|---|---|---|
| | United States | | |
| Rayonier, Inc. | Shelton, Wash. | 300 | 1945 |
| Eastern Corp. | South Brewer, Me. | 155 | 1947 |
| Eastern Corp. | Lincoln, Me. | 90 | 1948 |
| Crown Zellerbach Corp. | Lebanon, Ore. | 55 | 1949 |
| Wausau Paper Co. | Brokaw, Wis. | 90 | 1950 |
| Consolidated Water Power & Paper Co. | Wisconsin Rapids, Wis. | 140 | 1951 |
| Rayonier, Inc. | Hoquiam, Wash. | 400 | 1952 |
| Columbia River Mills[a] | Vancouver, Wash. | 110 | 1952 |
| Scott Paper Co. | Anacortes, Wash. | 100 | 1952 |
| St. Regis Paper Co.[b] | Bucksport, Me. | 100 | 1953 |
| Scott Paper Co.[c] | Everett, Wash. | 310 | 1954 |
| | Canada | | |
| Fraser Co., Ltd.[d] | Edmundston, N. B. | 350 | 1953 |
| Restigouche Co., Ltd.[e] | Campbelltown, N. B. | 260 | 1954 |
| Irving Pulp & Paper[f] | Saint John, N. B. | 150 | 1954 |
| Canadian International Paper Co. | Gatineau, Que. | 180 | 1954 |

[a] Currently considering converting plant at Salem, Ore., to ammonia pulping.
[b] Currently running comparative tests.
[c] Converting in August, 1954.
[d] Converted on trial basis November, 1953; currently continuing on trial basis.
[e] Converted on trial basis June, 1954; currently continuing on trial basis.
[f] Extended trial scheduled to start September, 1954.

## AMMONIA PULPING AS PRACTICED AT LEBANON, ORE., PLANT OF CROWN ZELLERBACH CORP.

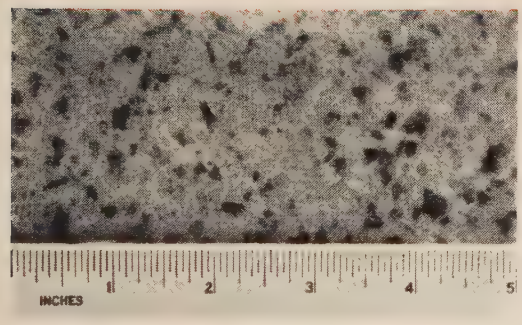
This plant was converted in December, 1949, as a joint venture by Crown Zellerbach and Soundview Pulp Co. The two companies constructed a pilot plant in 1951 to study evaporation and burning of the liquor and sulphur recovery. The pilot plant and its operation have been summarized (*6*). In 1953, Crown Zellerbach installed an evaporation, burning, and sulphur recovery system at the plant.

Crown Zellerbach Corp. operates pulp and paper mills in West Linn, Lebanon, and St. Helens, Ore., Port Townsend, Port Angeles, and Camas, Wash., Ocean Falls and Elk Falls, B. C., and Carthage, N. Y. Both kraft and sulphite processes are employed. The plant at Lebanon is one of the company's smallest and oldest operations. First known as O'Neil and Callaghan's mill, the plant was constructed from an old flour mill and produced its first pulp and paper products in 1890 (*18*).

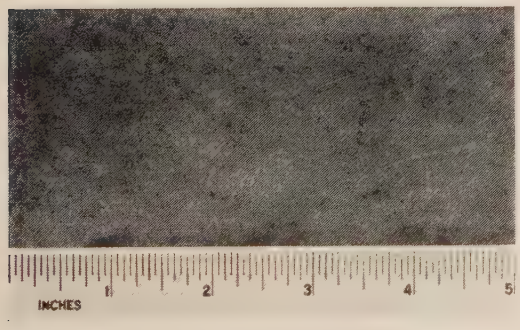
Of historical interest is the fact that until 1906, straw was used in papermaking in this plant. Waste straw was purchased from farmers for $3.50 to $4.00 a ton, and during that period, huge stacks of loose straw (often several blocks long and 50 to 60 ft. wide) piled high, dominated the Lebanon landscape. First prod-

**Less Horsepower per ton of pulp production**

# the Downingtown Fibrepulper utilizes power more efficiently . . . !




*High wet strength government map paper after 30 minutes pulping in water 200°F. in Downingtown Fibrepulper.*




*Same paper after 90 minutes of pulping.*

Because of its shape and design, the Downingtown Fibrepulper increases the number of times the pulp is passed through the defibring area within a given interval of time. This results in a more thoroughly refined pulp and a consequent appreciable decrease in kilowatt hours required per ton of output. With the high pulping rate the size of the unit is smaller for a given tonnage output. This reduction in size results in space saving and installation cost.

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uct from the mill was butcher wrapping paper. In a more esthetic vein, the pioneer papermakers were proud that some of their fine wallpapers, made from straw, found use in the White House in Washington.

The O'Neil-Callaghan mill became Lebanon Paper Co. and was subsequently purchased by Crown Zellerbach Corp. The plant has a rated capacity of 60 tons per day. Chief products include a special cartridge wrap for the explosives industry and butcher and merchandise wrap. The plant does not produce bleached products. Much of the plant's producing machinery and equipment has been in service many years, and numerous modifications have been made, some very basic. Those that have undergone major changes over the years are not listed in the usual manner in the processing equipment bibliography.

The mill is located on the south fork of the Santiam River in the Willamette Valley (approximately 85 miles south of Portland). Hemlock (90%) and white, fir (10%) represent chief woods used. These are bought on the open market on a grade and board foot basis. The mill issues purchase orders to its suppliers in the Spring so that each can anticipate his volume of wood to be delivered to the mill. Logs are brought to the mill by trucks from growing areas within a 70 mile radius. In earlier years, logs were received on a year-round basis. Within the past few years, however, use of wood chips from sawmill wastes has grown to 25 to 30% of the plant's wood raw material, resulting in a corresponding decrease in use of logs. Deliveries are now chiefly restricted to the period from Summer to late November. The chips may be used alone or blended with those from purchased logs.

Upon arrival at the mill, logs are inspected and graded. This is done by independent log graders who class logs in decreasing order of quality as grade 1, 2, or 3 on basis of many factors (number of knots, rot, soundness, etc.) as specified in applicable grading rules (*2*). Graded logs are lifted from trucks by an unloading crane which handles 35 loads per 12-hr. day during the heavy Summer and Fall seasons. Logs are bundled by loads and tied with no. 6 baling wire, inventoried, and towed by motor boat to the desired area of an 8 to 10-acre artificial storage pond. Each bundle or truck load contains approximately 5000 board ft. log scale (equivalent to slightly over 10 cords of wood). About 5,000,000 ft. (log scale) can be stored in the pond.

It is desirable to turn over the entire pond contents each year, but it is a "must" to do so every other year. "Cold-deck" storage is provided on the banks surrounding the pond. A heel boom crane mounted on a flat-bed railroad car lifts logs from the pond and stacks them for this additional storage which, in 1953, provided 7,000,000-ft. capacity.

In the wood mill, logs from the storage pond are sawed, barked, and split and then conveyed to the chipper for size reduction. From the storage pond, the logs are floated in bundles to the log haul-up, which takes the individual logs up an incline to the mill. Individual logs that have too high a water content, commonly called "sinkers," are identified as such so that when the logs are temporarily returned to the storage pond after sawing, sinkers will be routed directly to the mill for processing and not allowed to sink to the bottom of the pond.

The cutoff saw has a 72-in. diameter blade and is operated at 487 r.p.m. by a 50-hp. motor. It serves a double purpose: logs up to 48 in. in diameter are cut to 8 to 12-ft. lengths for the barker; and after barking, the logs are cut by the same saw to 4 or 5-ft. lengths.

Wood bark has little or no pulp value. It also is highly colored and contains dirt and other extraneous matter. All must be removed before the conversion to high quality pulp. Older methods of barking were by friction through tumbling or rotating the logs in a drum or in a stationary machine with agitating cam equipment. Hydraulic barkers are more common today. The hydraulic barker at Lebanon was converted from an old plane barker. A traveling arm moves at 1 ft. per sec. lengthwise of the logs. Water, at 1250 to 1350 p.s.i., is directed on the log by jets ($^5/_8$ by $^3/_{16}$-in. nozzles) mounted on the traveling arm. These jets reciprocate in a direction at right angles to the logs. After getting a pass down its length, the log is rotated 90°, and the arm travels back in the opposite direction and barks a second quadrant of the log. In two full trips, the barker generally removes all of the bark present. The barker uses approximately 200 gal. of water per min.; it takes 1 min. to bark a 12-ft. log. The machine is generally operated for two shifts a day.

The problem of bark disposal must be considered by every pulp mill. Depending on the type of wood, success in utilization has varied. Bark from southern pine has been used in making roofing felt. Spruce bark has been used in the manufacture of board and cheap grades of wrapping paper. Insulating materials have been made from finely shredded balsam fir and spruce barks. Potentially, some barks are a source of wax.

The most common disposal is as fuel. The heating value of moist bark (based on dry bark heating value of 8500 B.t.u. per lb.) ranges from an effective B.t.u. content of 7250 to 5580 with a range in water content of 50 to 70% (*12*).

At Lebanon, bark is routed to the hog fuel pile (sawmill waste) for burning. The plant uses approximately 120 units of 200 cu. ft. each of hog fuel daily.

From the hydraulic barker, logs are conveyed to the saw previously described, cut into 4 or 5-ft. lengths, and then split into four or more pieces. A horizontal, steam-driven (125 p.s.i. steam) splitter is used. The wedge-shaped, free-floating, steel cutting blade, 8 in. thick and weighing 200 lb., is driven partially into the horizontal log in the 48-in. stroke of the piston, splitting the log into halves; the trunnions on which the logs rest rotate the logs 90°; and the halves are split into quarters. On logs of large diameter it may be desirable to repeat the operation. The splitter has a capacity of 50,000 ft. in an 8-hr. shift.

Some of the wood pieces contain areas of bark not removed by the hydraulic barker. This is usually around knots or other uneven areas. A hand-operated cleanup barker is used to remove such bark when present. It consists of a 30-in. revolving disk containing four knives. The disk rotates at 567 r.p.m. as the operator holds the block against it, and the bark is shaved off as the operator revolves the block.

The clean, barked wood is loaded by hand into flat cars on a narrow-gage track and towed to the chipping machine in the main mill building.

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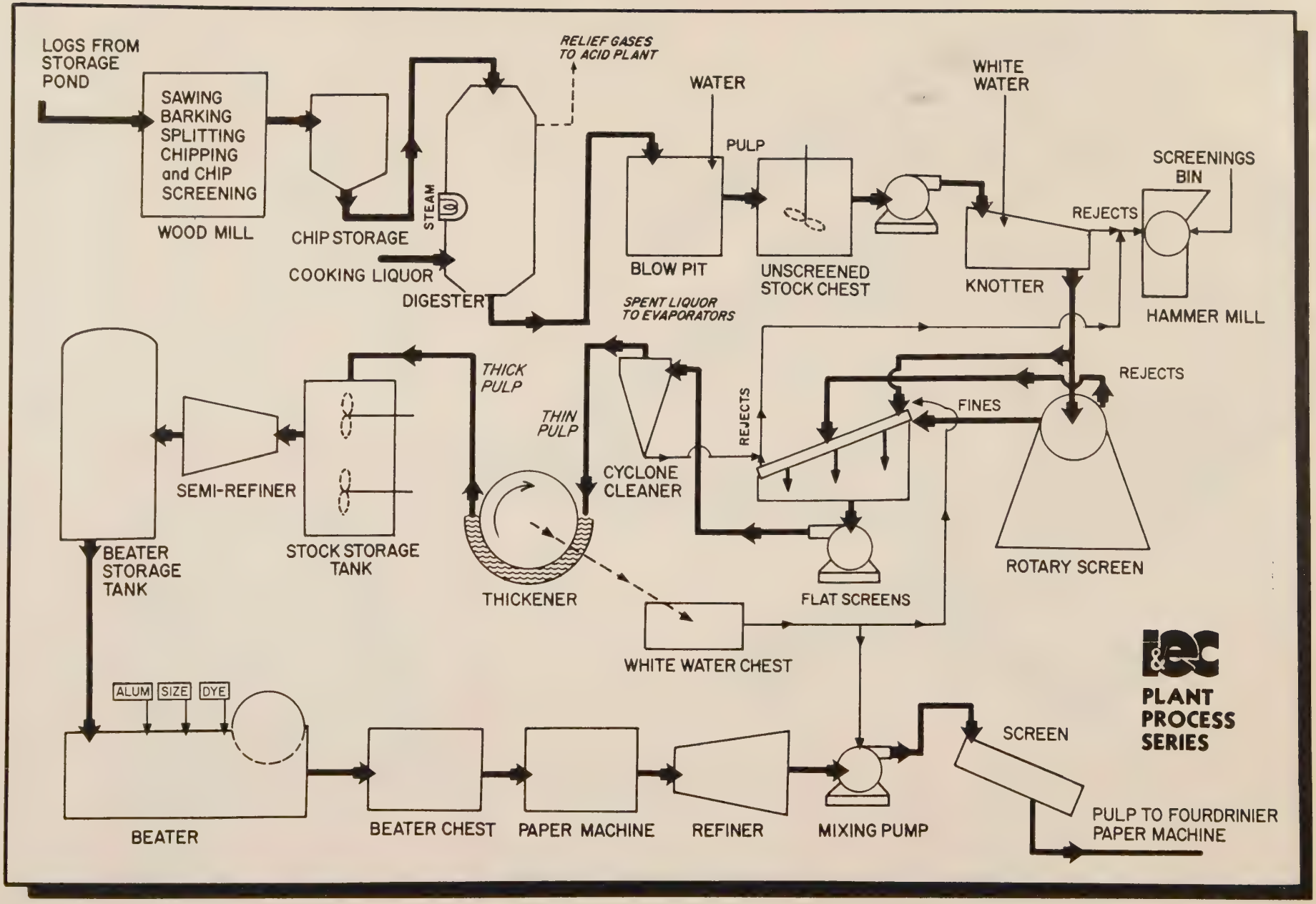


*Fig. 1. Flow sheet for cooking, screening, and preparation of pulp, Lebanon, Ore., plant, Crown Zellerbach Corp.*

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### *Chipping*

In order that the cooking liquor may penetrate the wood quickly, completely, and uniformly, it is necessary that pieces from the wood mill be reduced to chips averaging $^5/_8$ to $^3/_4$ in. long and $^1/_8$ in. thick. Multiknife machines, containing from 3 to 12 knives, are generally used. The Lebanon chipper consists of a cast steel disk 84 in. in diameter, which is rotated at 600 r.p.m. by a 100-hp. synchronous motor (*16E*). The disk contains three hard carbon-steel knives 22 in. long. A 17 by 17-in. feed spout is inclined at an angle of 57° to the rotating disk and knives. An operator uses a sharp hook or pickaroon to pull a piece of the wood from the flat car and drop it into an inclined chute that feeds into the chipper spout. The wood is chipped as it comes in contact with the rotating knives. The chips pass through slots behind the disk knives into a pit below. The chipper has a capacity of 10,000 board ft. per hr. On Pacific Coast woods, the knives must be sharpened after 4 to 6 hr. of use.

Generally, wood from chippers contains from 1.5 to 5% sawdust and pin chips and 1.5 to 3% slivers. Chip screens are used to separate the good chips from the under- and oversized particles. In some plants, circular and shake-type screens are used. Crown Zellerbach uses a double-deck vibrating screen (*18E*). The upper screen contains $1^3/_8$-in. holes on $2^{13}/_{16}$-in. centers, and the lower $^1/_4$-in. holes on $^3/_8$-in. centers. Fines (1 to 2% at this plant) are routed to the boiler room; slivers, which vary widely in quantity, are recirculated to the chipper.

Wood chips purchased from sawmills are dropped from railroad cars (holding 17 to 20 units) to conveyors which discharge to the chip screens. These are subsequently processed the same as those produced at the plant.

Chips of proper size are conveyed to chip storage bins which are located above the digester charging floor.

### *Digesters*

In production of pulp (Fig. 1), the lignin and other undesired components of the wood are separated from the cellulose fibers in the cooking process.

Digesters in the industry are sized from a minimum of 10 tons to nearly 30 tons capacity. At Lebanon, Crown Zellerbach has three stationary digesters. They are 40 ft. in over-all height, approximately 15 ft. in diameter, and capacities range from 3700 to 4275 cu. ft. Each is fitted with a bell top and coned bottom. In the top is the charging opening; in the bottom, the discharge valve. The cylindrical center section is constructed of $1^1/_8$-in. steel plate. Each digester has an external, stainless steel (type 316) heater (steam on outside) containing 90 tubes $1^1/_8$ in. in diameter and 12 ft. long (300 sq. ft. of heating surface). Linings before conversion from calcium bisulphite pulping were of standard acidproof brick with litharge-glycerol joints. These linings were replaced as needed with others especially designed for ammonia bisulphite liquor. Special features of the new linings are double-course, high density, acid-resisting brick with furfural-phenol jointing and with a membrane of the same material between the two courses. These have proved satisfactory.

Cooking cycles vary from 6 to 12 hr. In a typical 12-hr. cycle currently used at Lebanon the procedure is as follows:

Chips (18 cords of wood, which makes 9 tons of pulp) are fed from the overhead bins by gravity through the charging hopper into the digester. Flow and packing of chips is aided by steam jets on the charging hopper. Ammonium bisulphite cooking liquor (Fig. 3) containing an excess of sulphurous acid (preparation of which will be subsequently described) is added at the same time from the bottom. Cooking acid temperature is 86°F.; it contains a total of 6.50% sulphur dioxide (0.94% combined, 5.56% free). Approximately 21,000 gal. of the liquor is required to cover the chips. Filling the digester requires about 30 min. The lid is then secured. Two systems of steam are provided. During the first 8 hr., indirect steam (10 to 30 p.s.i., in the heater) is used. For balance of the cooking period, direct steam is admitted ($3^1/_2$ hr).

Cooking liquor is circulated from the bottom of the cylindrical section to top and bottom of the digester through the heater by a centrifugal pump. As the cooking cycle progresses, pressure builds up because of sulphur dioxide from the cooking liquor as well as the presence of air in the chips. When it reaches 80 to 85 lb., pressure is released through the top of the digester. Later in the process, pressure is "side relieved," gases and liquor being used to fortify the cooking liquor (Fig. 2). Temperature reaches a maximum of 275°F.

After the digester is closed and cooking begins, time-temperature-pressure charts from a typical cook show the following data:

| *Time, hr.* | *Temp., °F.* | *Pressure, p.s.i.* |
|---|---|---|
| 0 | 86 | 0 |
| 1 | 100 | 31 |
| $1^1/_2$ | 120 | 42 |
| 2 | 150 | 62 |
| $2^1/_2$ | 180 | 85 (top relief) |
| 3 | 187 | 85 |
| $3^1/_2$ | 194 | 85 |
| 4 | 205 | 85 |
| $4^1/_2$ | 207 | 85 |
| 5 | 211 | 85 |
| $5^1/_2$ | 222 | 85 |
| 6 | 229 | 85 |
| 7 | 244 | 78 (side relief) |
| 8 | 267 | 71 |
| 9 | 272 | 40 |
| 10 | 269 | 29 |
| 11 | 262 | .. |

Toward the end of the cooking cycle, sulphur dioxide of the cooking liquor is determined by standard NaOH–$KIO_3$ titration (Palmrose method). Desirable final sulphur dioxide content is 0.4%. Samples are also compared with a set of color standards; when desirable color is reached, indicating completeness of cook, pressure is reduced to 30 p.s.i. The digester is blown at this pressure to a blow pit below.

There are three wood blow pits, each of which holds about 12 tons. Contents of the digester are discharged into the pits through 12-in. blow lines. Wood "target" blocks are mounted on the side of the tank opposite the blow lines, and the digester contents are directed toward the targets to break up pulp masses. Usually, 2 ft. of 10% spent liquor (7000 gal.) is added to the blow pit before blowing to cushion the pulp.

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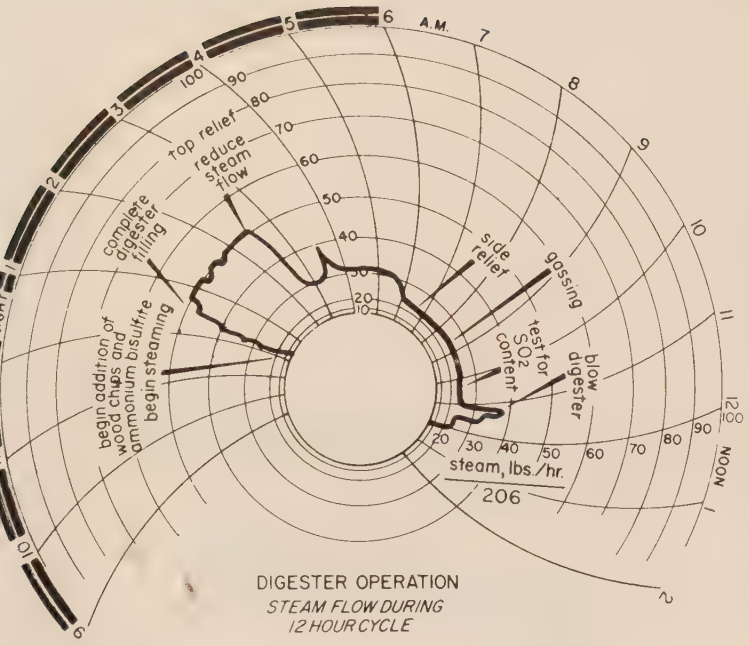



*Fig. 2*

Operation of the digesters or cooking process if favorable when compared with calcium bisulphite cooking. Good pulp is produced in cycles as short as 6 hr. It is very difficult to produce other than inferior pulp in 6 hr. by calcium base. Generally the minimum time for calcium base would be about 7 hr. Longer cooking cycles are generally practiced when operations permit. A 12-hr. cycle, as described above, is unusually long for United States practice. When lower temperatures can be used, effect on pulp is less drastic, and a better yield of stronger pulp is obtained. The pulp is also brighter and more easily bleached. At the same acid composition, rate of cooking in ammonia base is slightly more rapid than with dolomite. With ammonia base, however, combined sulphur dioxide can be lowered drastically without deteriorating the quality of the pulp. At lower combined sulphur dioxide, the rate of the cooking reaction is considerably increased and the cooking time or temperature can be lessened accordingly. (In test runs, cooking temperature has been reduced from 286 to 266°F. without changing the time of cooking.)

As mentioned, liquor color (along with chemical analysis) is a guide for blowing the digester. On change-over to ammonia pulping, it was necessary to establish a new set of standards as the liquor is considerably darker for any degree of cooking or delignification. Operators have found that color of the ammonia-base acid is a more reliable indication of degree of cook than it was when the calcium base was used. Generally speaking, the cooking procedure has proved more flexible and the pulp less subject to damage from improper treatment than they were before conversion.

### *Screening*

The digester charge which has been discharged to the blow pit consists of the insoluble cellulose fibers together with the spent liquor containing all the soluble wood constituents. These must be separated before the pulp can be further processed. Rich liquor (containing 10 to 11% solids) is drained through a perforated stainless steel (type 316) drain in the bottom of the blow pit to an outside storage sump. The blow pit is then washed, first with weak wash liquor and finally with water.

At a predetermined temperature (131 to 140°F.), which corresponds to a solids content of 9 to 10%, an air valve is automatically thrown, and the wash liquor is transferred to a weak liquor storage tank. At a second predetermined temperature (110 to 120°F.), which corresponds to a solids content of 1 to 2%, a valve transfers the weak washes to a sewer. All washes discharged to the sewer pass over a flume where samples are taken. Dissolved oxygen in the river is not allowed to fall below 5 p.p.m. (The disposition of spent liquor is described in the section of this report dealing with evaporation and recovery.)

In a larger or more complete installation for spent liquor recovery from the pulp, it is probable that rotary vacuum washes would be used.

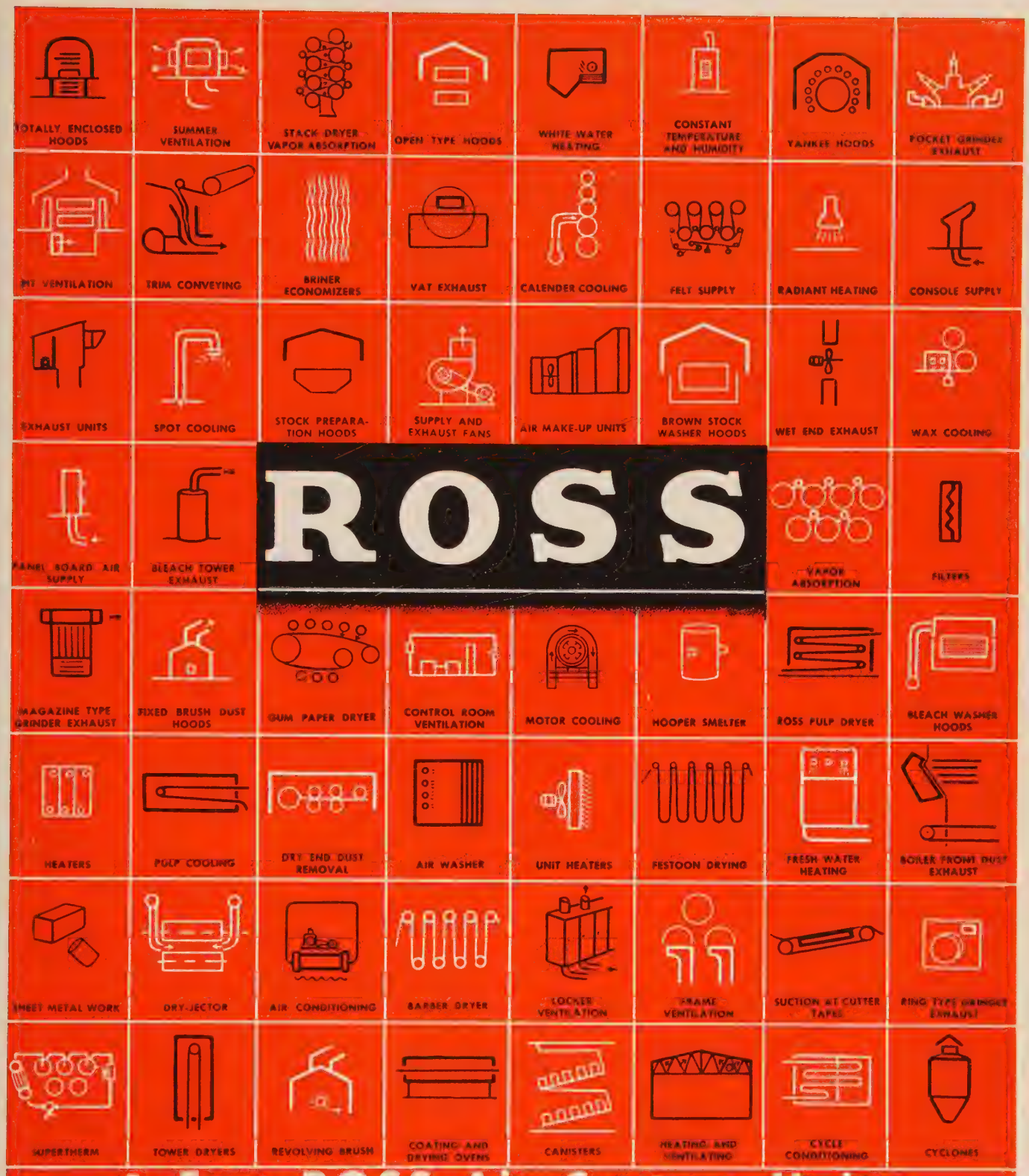
It is important to remove as much of the cooking liquor as possible. Usually three washes are made, requiring $3^1/_2$ hr. The pH of final wash liquors is maintained at a minimum of 6 by addition of water. Lower pH's cause foaming.

The washed pulp is sluiced by gravity and addition of water to the unscreened pulp chest. This is an impeller-agitated concrete tank (capacity of 4800 cu. ft.) divided into two channels by a (mid-feather) or central wall. From here it goes to the feedbox ahead of the knotter. The consistency is maintained at 3 to $3^1/_2$% through addition of water (*5E*).

One of the most important operations in the treatment of pulp is separation of uncooked slivers and knots from the cooked fibers and removal of dirt and other foreign matter. Various types of screens are used for these purposes; generally, the operations are divided into coarse and fine screening. In all, water plays an important role, providing both a medium for screening as well as a method of conveyance between screens. After screening, water, commonly called "white" water, is extracted and reused. At Lebanon, knots and oversized particles are removed by a vibrating screen containing $^1/_8$ by $^3/_4$-in. slotted opening (*1E*). Tailings are passed to a hammer mill revolving at 1800 r.p.m., and discharge is used in certain coarse grades of paper (*9E*). White water is added at the knotter, reducing consistency of the pulp to 0.2 to 0.25% in order that fine screening can be accomplished.

Typical pulp mill flat and rotary screens are used for removal of small knots, fiber bundles, and dirt; final cleaning is by cyclone-type cleaners. Flat screens consist of chrome-plated screen plates, 12 in. wide, 43 in. long, and about $^3/_8$ in. thick, containing slots ranging from 0.006 to 0.010 in. in width, usually referred to as 6-cut, 10-cut, etc. Plates are mounted in the same plane slightly inclined to the horizontal in a long trough. Below the plates are vibrating rubber diaphragms. The screen depends for its operation on a combination of gravity flow and suction from the diaphragms.

Rotary screens vary widely in design and operation. Some types consist of either fixed or stationary horizontal perforated cylinders with rotating paddle wheels in the center. Stock is fed into one end of the cylinder and is forced along and through the screen section which may or may not be graduated. Accepted stock passes through the screens.

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At the Lebanon mill there are 10 flat screens, each consisting of 12 plates. Five screens are in each of two lines arranged in U-formation. The slots run from 6 to 12-cut, the plates having the larger slots being near the receiving end, where the stock flows heavy; those having smaller slots are at the opposite end, where flow of stock is lighter and dirt is more concentrated. All screenings from flat screens are returned to the hammer mill after the knotter; total feed to the hammer mill is 3% of the feed.

There is also a rotary screen (*21E*) with 0.055-in. perforations. Rejected stock from the centrifugal screen is pumped to the second section of the flat screens. Accepted stock from the centrifugal screen is combined with the accepted stock from the flat screen and pumped through a series of cyclone-type cleaners. These consist of primary and secondary units which remove fine dirt left in the pulp in first screening operation (*2E*).

The Lebanon mill has experienced many advantages in screening since converting to ammonia pulping. The pulp produced here is a hard pulp (TAPPI permanganate no. 18), and formerly 4 hr. were required to fine screen a cook. As a result of better penetration of the ammonium bisulphite cooking liquor and more uniform and thorough cooking, screenings have been decreased 50% and screening time to 3 hr. This has been accomplished in spite of the fact that there was a redistribution of screen plates and that more fine-cut plates were installed to screen out dirt from ingrown bark around knots that was freed as result of better penetration of the ammonium-base acid.

***Conversion of Pulp to Paper***

To simplify presentation, further treatment of the pulp and processing to finished paper product, which is to a great extent standard with other sulphite mills, is treated briefly in this report. The more unique acid preparation and evaporation and use of cooking liquor are then described.

Pulp from the cyclonic cleaners contains only one material to be removed—water. The consistency is only 0.1 to 0.2%. This is increased to $3^1/_2$ to 4% on a stock thickener (*4E*).

A thickener is essentially a wire mesh-covered cylinder rotating in a vat containing the thin screened stock. The pulp adheres to the rotating screen surface while the water passes to the inside of the cylinder and flows to the white water tank. The thickened stock discharges from the opposite side of the vat and is pumped to the stock storage tank. This is a reinforced-concrete tank which holds 21 tons of 3.8% pulp (moisture-free basis). The tank has a tile mid-feather, and circulation is provided by impellers in both the upper and lower parts of the tank.

From the storage tank, the pulp is pumped through a refiner (*13E*), which gives the pulp a preliminary mechanical treatment, to the beater storage tank ahead of the beaters. To this point, pulp processing has been continuous; some batch operations follow, the first of which is beating.

Beating has been considered to be a major part of the transition between pulp and paper. Various types of equipment have been used in the industry for beating, during which the various components of the furnish are added, the pulp is hydrated, and the surface character of the fibers is changed. The expression, "paper is made in the beater," has long been associated with the functioning of the Hollander beater, which was first used. However, many mills have substituted other types of equipment to perform this operation.

At Lebanon, hydration of the pulp occurs in the refiner. The beater is principally used to blend the added ingredients for papermaking with the pulp. This arrangement is peculiar to this mill only. Hollander-type beaters are used—three for regular pulp and one for repulping "broke," or trim, from the paper machines. The machine consists of an oval-shaped tub which holds 1200 lb. of stock (*10E*). A partition extends lengthwise through the center and at one end is a horizontal roll, 55 in. in diameter, with bars, 72 in. long, laterally positioned. The roll rotates in the pulp at 140 r.p.m. and engages a bed-plate arrangement at the bottom of the tub, permitting bar-to-bar action between it and the roll. Thorough mixing and agitation are thus provided as the pulp is circulated about the tub and through the bars.

The kind of paper being made determines the type and amount of nonfibrous raw materials to be added to the furnish. For the products at Lebanon, rosin size and papermakers' alum are added to all and aniline dyes to certain colored products. The size is added to make the paper partially water repellent; the alum, $Al_2(SO_4)_3 \cdot 18H_2O$, precipitates the size on the pulp and sets certain colors through adjustment of the pH.

In a typical beater batch, 1200 lb. of stock is first fed to the beater, which takes approximately 1 min. Size ($1^1/_2$ to 4%) is added, followed by addition of the same amount of alum. The batch is then mixed for approximately 30 min. and discharged into a rectangular tile-lined beater chest in the basement. The consistency at this point is 3.5%. Water is added reducing it to 3.2%. From the chest, the pulp is pumped continuously to the paper machine chest in the paper machine room.

Before the pulp becomes paper, it once more undergoes refining. Standard jordan refiners are used at Lebanon (*11E*). The jordan consists of a horizontally positioned conical shell which contains a rotating, correlative, cone-shaped plug. From the jordan the thoroughly refined pulp is delivered to the paper machines by a pump to which white water is added, lowering the consistency to 0.5%. The thin stock is passed over a fine screen for removal of any dirt or heavy particles that may be present.

The balance of the conversion from pulp to paper occurs on the fourdrinier "wire" and fourdrinier paper machine (*14*). The entire process is one of aligning and interweaving the fibers together with dewatering to form the paper (sheets). Crown Zellerbach has two paper machines at Lebanon (*17E*, *20E*). One machine is capable of making finished paper 64 in. wide at speeds up to 450 f.p.m.; the second makes paper 76 in. wide at speeds up to 520 f.p.m. Of interest is the fact that the paper machine and its auxiliary equipment are fed with stock containing 99.5% water and the finished paper leaving the dry end contains 5 to 8% water.

***Acid Making***

The Lebanon plant used a conventional milk of lime

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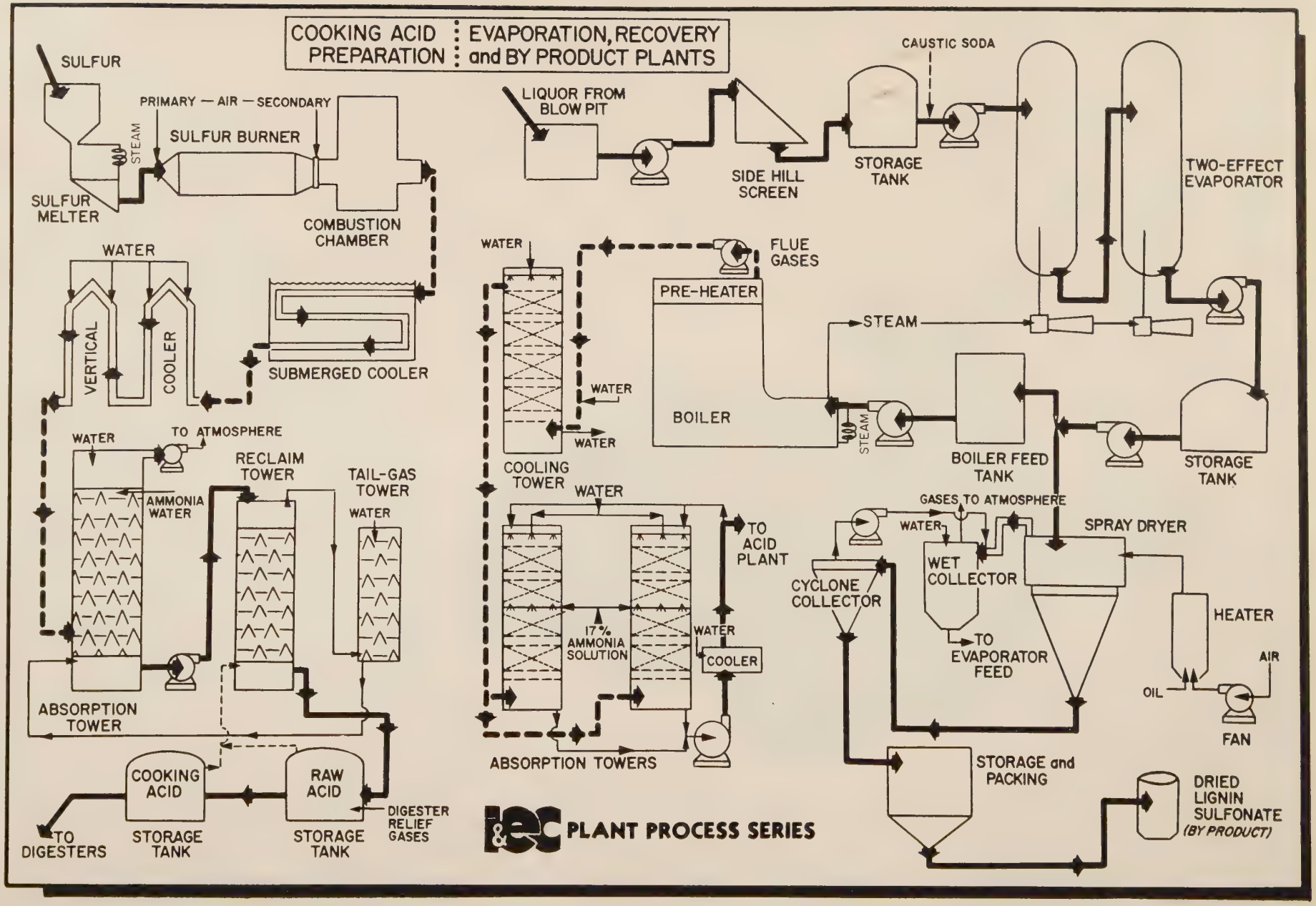
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*Fig. 3*

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Barker system (Fig. 3) when using a dolomite lime as a base. Raw materials for the Barker process include sulphur or pyrites, limestone, and water (*13*). Briefly, the Barker system consists of burning sulphur to produce sulphur dioxide gas, cooling the gas, and absorbing the gas in water in presence of lime (as milk of lime). This liquor or raw acid is calcium bisulphite. To it is added sulphur dioxide gas relieved from the digesters to obtain the fortified cooking acid. Major pieces of equipment include sulphur burners and combustion chambers, gas coolers, absorption apparatus, and equipment for strengthening the raw acid with digester relief gas. Conversion of a calcium-base plant to ammonia base is relatively simple and consists essentially of substituting an ammonia solution for the milk of lime in the Barker acid plant. At Lebanon, most of the old equipment is still used, and the principal addition to the plant is ammonia handling and storing facilities.

At Lebanon, sulphur is used as a source of sulphur dioxide gas. The sulphur is melted in a vat containing coils and pumped through a steam-jacketed line to the single sulphur burner. This is a conventional rotary burner, 4 ft. in diameter and 11 ft. long, equipped with a brick-lined combustion chamber 9 ft. in diameter and 12 ft. high (*8E*). A bubble-type liquid-level controller in the burner maintains the pond level within 0.25 in. and permits regulating the gas at 18 to 19% sulphur dioxide (*3E*). The gas leaving the combustion chambers is 1500 to 1600°F; it must be cooled so the sulphur dioxide can be absorbed in water. Rapid cooling is necessary in order to prevent the formation of sulphur trioxide. A pond cooler, in which the gas passes through pipes submerged in water and a vertical lead cooler, in which water is applied to the exterior of the pipes in which the gas flows, result in an over-all temperature drop to 70°F. The cooled gas passes through an absorption tower, where it is combined with aqua ammonia to form ammonium sulphite.

Ammonia is received as anhydrous ammonia in tank cars holding 50,000 lb. It is unloaded under its own pressure. As it flows through a pipe equipped with baffle plates, water is admitted to give a 17% aqueous solution. Rate of water flow is controlled by solution temperature rise which is proportional to the amount of dilution. A temperature controller with a temperature bulb in the solution actuates a control valve in the water supply. Storage tanks with capacity in excess of a full tank car (reduced to 17% concentration) are available. Aqua ammonia is pumped to the absorption tower by a 5-g.p.m. gear pump with a hydraulic, variable-speed drive; a flow of 2 g.p.m. is used, which is checked by a recording rotameter.

The absorption tower is $3^1/_2$ ft. in diameter and 43 ft. high. It is divided into two sections, each of which is packed with staggered 3-in. spiral tile. The bottom section is 27 ft. high. At the top of this section, water (80% of total) and the aqueous ammonia solution are added to the raw acid gas, which is admitted at the bottom of the lower section. Ammonium bisulphite and free sulphur dioxide solution are formed. Balance of water (20%) is added at the top of the upper section of the column to safeguard against losing any ammonia; this has worked satisfactorily as the water scrubs out the final trace of ammonia. Gas is pulled through the system with a stainless steel vacuum pump (*14E*).

The raw acid leaves the bottom of the absorption tower at 60 to 70 g.p.m. It contains 2.8 to 3% sulphur dioxide—1.3% combined $SO_2$, the amount of sulphur dioxide that would be contained in $(NH_4)_2SO_3$ or "papermakers" sulphite. The raw acid is pumped to the top of a 4 by 20-ft. wood reclaim tower. Fifteen feet of this tower is packed with 3-in. spiral tile. The acid flows from the bottom of the tower to a 25 by 18-ft. (62,500 gal.) storage tank. Here the sulphur dioxide relief gases from the digesters, after passing through a tubular heat exchanger (*6E*), are bubbled up through the acid, building up the total sulphur dioxide content from 3 to 6.5 to 7%. The gases not absorbed in the mixing tank go to the bottom of the reclaim tower. The fortified cooling liquor is pumped to the cooking acid storage tank, from which it is available for use in the digesters.

The gases discharged from the reclaim tower go to the bottom of a 14-in. diameter by 15-ft. high tail-gas tower packed with $1^1/_2$-in. Berl saddles. Water (8 to 10 g.p.m.) is added to the top of this tower, removing the last trace of sulphur dioxide. This goes to the bottom of the absorption tower.

Since conversion to ammonia pulping, the acid plant is almost an automatic operation. Recorders are installed on the water flow to the towers and one on the vacuum after the coolers and after the towers. Temperature and sulphur-dioxide recorders are on the sulphur burning system in addition to the liquid level control.

Before conversion, the plant replaced all acidproof bronze valves and fittings with stainless steel parts. This completed a program already under way to replace bronze parts with stainless steel as the former corroded out. Maintenance of the acid plant has been materially simplified. In addition to elimination of scale and sludge in the cooking and acid-making systems, dust, characteristic of the dolomite operation, is no longer present.

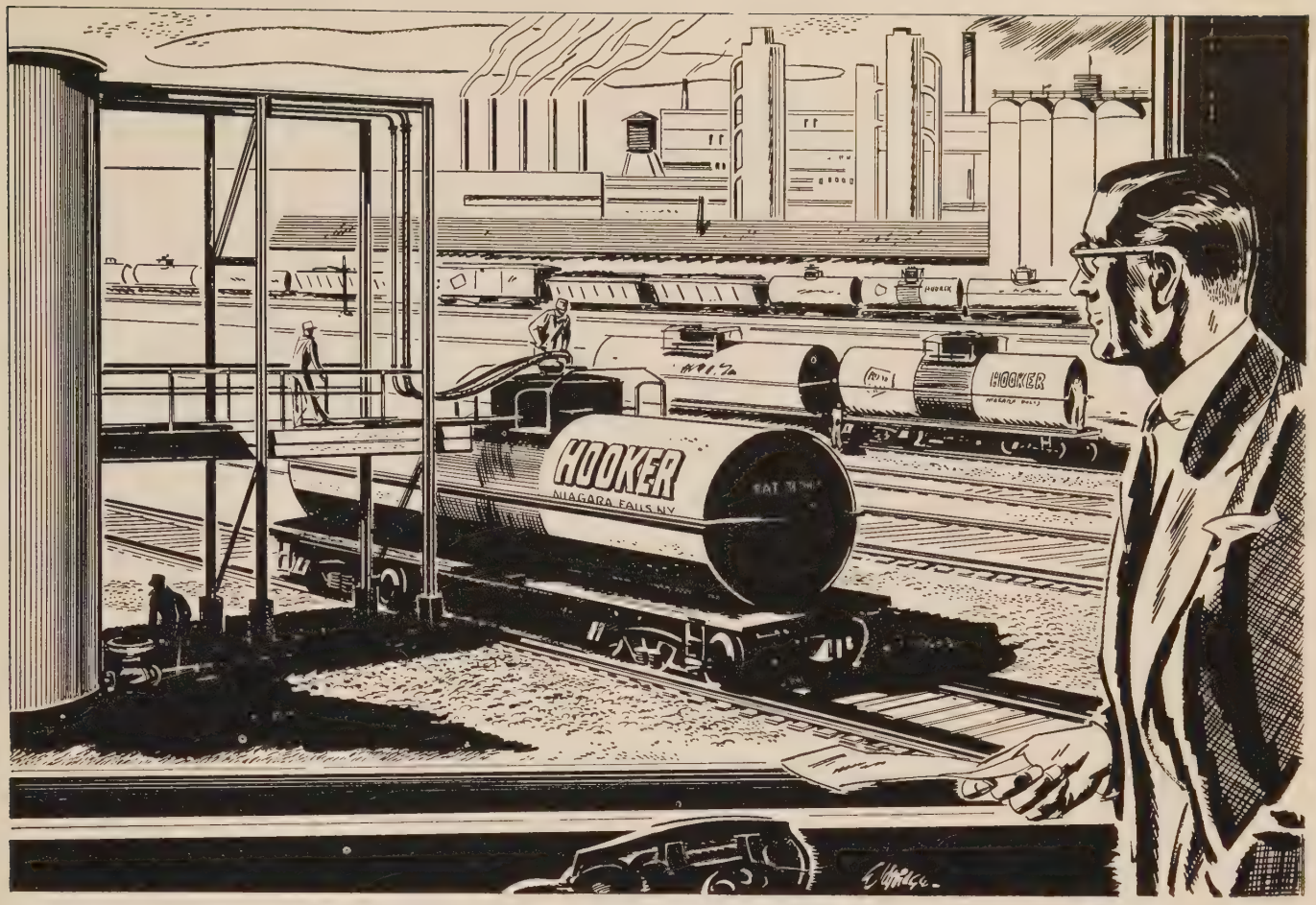
#### *Waste Liquor*

Following operation of an evaporation, burning, and recovery pilot plant in 1951, Crown Zellerbach installed facilities for carrying out these operations on a plant scale in 1953. Some of the results of the pilot plant operation have been published (*1*, *6*).

In the evaporation, burning, and sulphur recovery system (Fig. 3) spent liquors from the digesters are concentrated from 9 to 10% solids to 50% solids, burned under boilers, and sulphur dioxide in the flue gases is partially recovered by scrubbing.

The liquors from the evaporator feed sump are first pumped to a side hill screen or "saveall" which is simply a fourdrinier wire converted into a screen inclined at 60° where remaining fibers are removed. The liquor flows to a 50,000-gal. storage tank. Another pump transfers it to the evaporator.

The concentrating plant consists of a two-effect evaporator and auxiliary equipment (*7E*). The last effect is followed by a barometric condenser. The heaters in both effects are flat-plate heaters containing 2200 sq. ft. of heating surface. The first or high temperature effect operates as a falling film type. Operating pressure is 30 to 40 p.s.i. Circulation over plates is 30 g.p.m. Condensate is circulated on the steam side

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of plates at 40 to 50 g.p.m. Concentration of liquor is increased from 14 to 15% to 50% solids.

The second effect employs natural circulation. It is built exactly the same as the first effect but has a larger vapor head. There is no pump for falling film on the vapor side. The vapor passes to the barometric leg equipped with steam jet condenser, which can operate at 27 in. vacuum when necessary. The condensate from the first effect flashes to the second effect.

The entire evaporation system is piped so that the Rosenblad method of channel switching of liquid and steam plates for scale control can be employed. While the use of ammonia base eliminates scale forming materials from the cooking chemicals, there is enough calcium from the wood ash to warrant the use of this expensive channel switching equipment. Channels are switched every 8 hr. on the high temperature effect and every 24 hr. on the low temperature (25 pairs of valves, from $^1/_4$ to 16 in. must be changed each time). All plate materials coming in contact with the liquor or vapor are of type 316 ELC or 317 ELC alloy stainless steel.

It has been found with ammonia-base spent liquors that a concentration above 50% solids is not desirable. At best, there is a danger of increased viscosity which makes the liquor harder to handle and at worst, there is the possibility of an irreversible jelling due to polymerization. Some of these conditions have been reported in some detail (*6*).

The concentrated liquor goes to a 15,000-gal. storage tank from which it is pumped to a 1000-gal. wood stave tank that supplies the two boilers under which the liquor is burned. The latter tank is equipped with steam coils that maintain the heavy liquor at 190 to 200°F. The liquor has a heat content of 8800 B.t.u. per lb. on a solid dry basis. As fired, it contains approximately 4500 B.t.u. per lb.

A positive displacement pump, which operates at 100 to 150 p.s.i., moves the heavy liquor to the burners through stainless steel pipe and metering equipment (*23E*). The burners through which the liquor is fed to one of two boilers are conventional steam atomizing burners for high pressure oil except that the liquor is on the inside and the steam on the outside; liquor discharges through a central opening and atomizing steam enters through several radial openings at right angles to it. Liquor flow to both burners is regulated by flow recorder controllers.

In the original pilot plant study, an old horizontal water tube boiler (in which the gases flow parallel to the tubes) with a Dutch oven ahead of it was used. This is one of the two boilers now used in the regular plant operation. Grates were removed, and some brickwork was added to give a furnace of the proper dimensions. It is equipped with a steam preheater, a forced draft fan, an induced draft fan, and dampers for combustion control. The burner is mounted in a register in the center of the front wall of the Dutch oven. Preheated air (300°F.) from the heater enters through the register in a whirling motion imparted by louvers. Flue gases from this boiler go to the sulphur dioxide recovery system when it is being used.

The second boiler has no air heater and is not equipped for sulphur recovery. It is equipped with a Dutch oven for hogged fuel burning and with facilities for burning oil. It is necessary, however, to keep a hogged fuel fire going in the Dutch oven inasmuch as the combustion chamber, where the liquor is sprayed in, has water walls. Liquor is burned at a constant rate, and the swings in steam demand are taken up by varying the hogged fuel feed. This boiler also accommodates fluctuations in concentrated liquor supply when the waste liquor is being disposed of in some other manner, which is described later. Each boiler handles approximately 50% of the liquor when all of it is burned.

As mentioned, one of the boilers is equipped with sulphur recovery facilities. The system consists of a cooling tower and two absorption towers which operate in parallel (one of the latter was used as a cooling tower in the pilot plant).

The cooling tower is of wood stave construction, is 8 ft. 4 in. diameter and 35 ft. high, and is packed with 15 ft. of 3-in. spiral tile. It cools the hot gases from the burners at 450°F. to 100 to 105°F. Cooling water (70 g.p.m.) is added at the top of the tower and discharges from the bottom at about 200°F. Little sulphur is lost at this temperature.

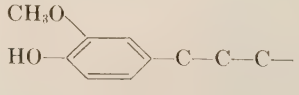
The two wood stave absorption towers (5 ft. in diameter by 34 ft. high) are packed with two 5-ft. sections of 3-in. spiral tile. An absorption solution of ammonium sulphite-bisulphite circulates over the bottom section and is pumped through a stainless steel heat exchanger after each pass over the packing, the circulating solution containing 0.5 to 0.7% sulphur dioxide. A 17% solution of aqua ammonia is added at the suction side of the recirculation pump; feed is regulated by a pH recorder controller. Make-up water (10 to 15 g.p.m.) equivalent to the acid sent to the acid plant is added above the top section. This water removes any ammonia stripped from the scrubbing solution. A portion of the acid from the tower is taken just after the heat exchanger and sent to the acid plant where it is added to the water going to the absorption tower.

Under proper operating conditions, about 80 to 85% of the sulphur in the liquor fired to the furnace is recovered; enough steam is obtained from burning the liquor to do the evaporating. Sulphur consumption normally runs from 275 to 280 lb. per ton of pulp produced. In plants producing bleaching grades of pulp, the consumption is 240 lb. per ton.

The spent liquor is also used on streets and roads for dust control by the city of Lebanon and a number of logging companies. During the summer, this outlet represents 10 to 15% of total spent liquor production. It is also used in by-product production.

#### *By-Products*

In 1952, Crown Zellerbach announced a new commercial product produced from the concentrated spent sulphite liquor which consists of a mixture of ammonium lignin sulphonates and sugars. The structure of the lignin sulphonic acids is not known; however, it is generally believed that they are composed of repeating unit polymers of guaiacyl propane




and have an average molecular weight of 5000. The sulphonic acid group is known to be attached to an ali-

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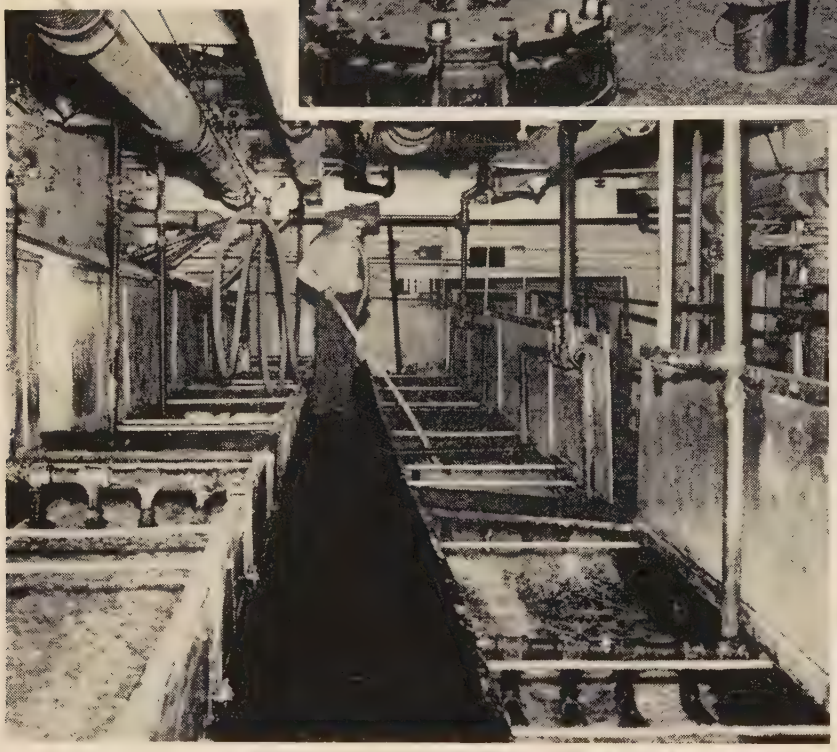
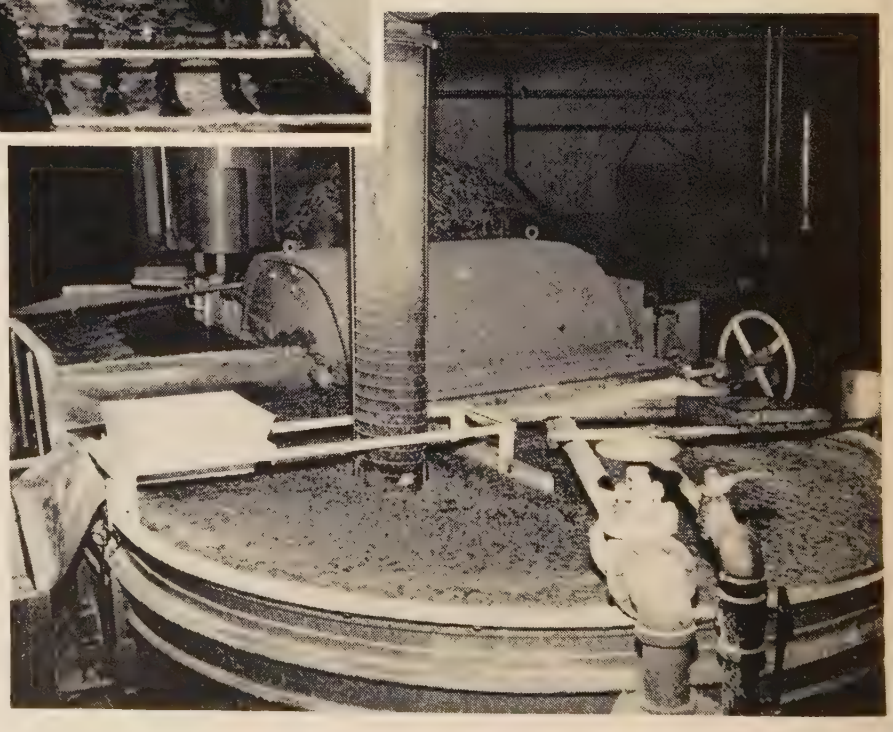
*Chips are charged from overhead storage bins to one of three digesters; on charging floor, only tops of units, 40 ft. high, are visible; charging hopper is at left*

## Ammonia-Base Pulping

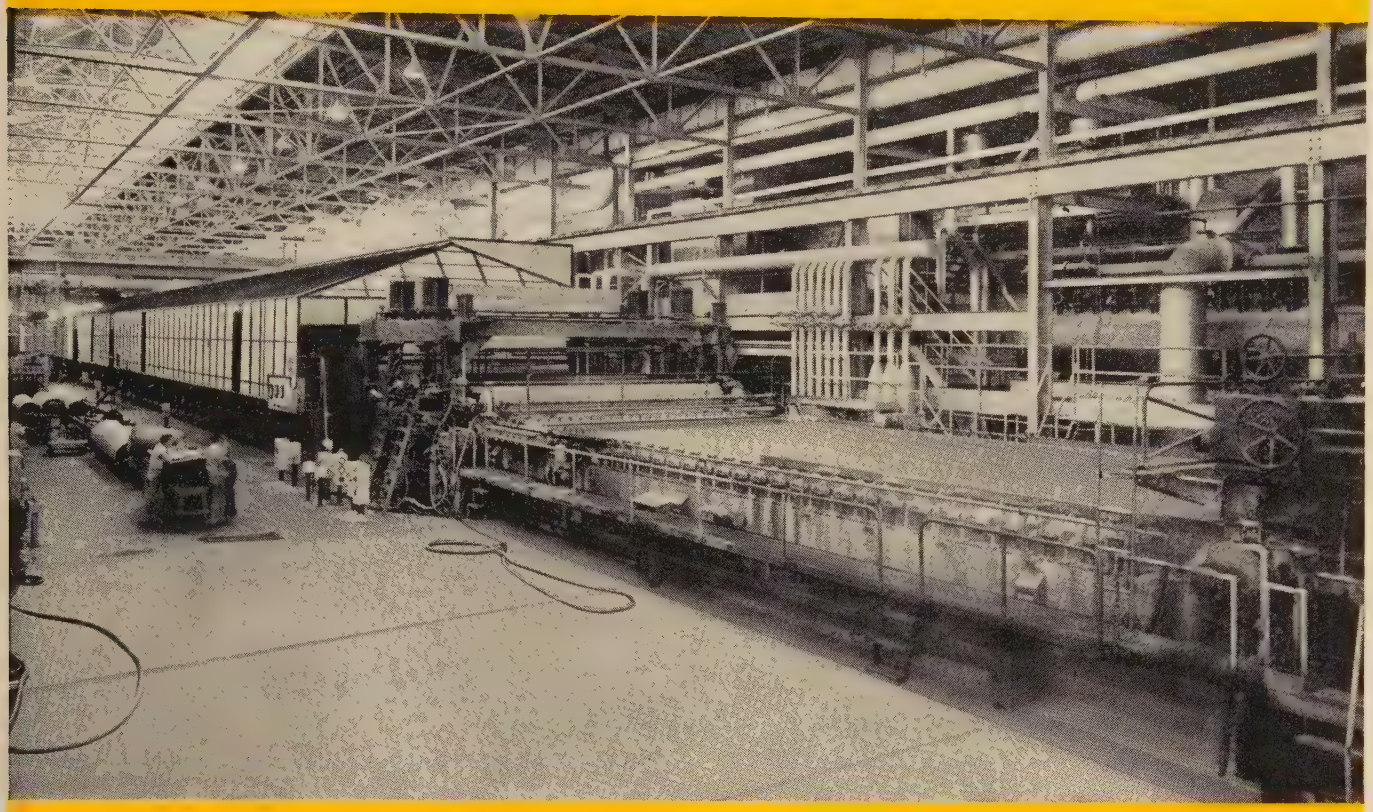


*Typical flat screens, which depend for their operation on a combination of gravity flow and suction from diaphragms, are used to remove fine knots, fiber bundles, and dirt after the pulp has been coarse screened*



*Size, alum, and dyes are added to pulp in a Hollander-type beater; at Lebanon very little hydration occurs in the beater, which is usual in other plants*

# *Union Bag with Puseyjones Machines*



New No. 6 Puseyjones Fourdrinier Machine at Union Bag and Paper Co., Savannah, Ga. Fourdrinier is of Rapi-Drape construction for 120-foot wire; Press Part of Simplex design with two suction presses; Dry Part comprises 71-60" paper dryers and 16 felt dryers, with intermediate smoothing-marking press and size press; eight roll calender stack; Puseyjones horizontal track reel for 96" rolls. A General Electric multiple generator electronic-controlled sectional drive provides power for the various machine sections.

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phatic carbon atom, probably adjacent to the benzene ring. It is also believed that one or more $OH^-$ groups appear on the three-carbon side chain (*4*).

The material, now available in a number of grades, is produced by spray drying the evaporator discharge. The material is a light-brown powder, the particle size ranging from 0.004 to 0.006 in. diameter. It is soluble in water in all proportions and has a pH of about 3.5 in a 20% solution. Heat value is 9100 B.t.u. per lb. (moisture-free basis). Water content is 3%.

The constituent analysis of the product is:

| | *Total solids, %* |
|---|---|
| Ligninsulphonic acids | 55.0 |
| Reducing sugars (as glucose) | 16.7 |
| Alkali-liberated $NH_3$ | 2.8 |
| Sulphate (as $SO_3$) | 1.6 |
| Sulphated ash | 2.0 |

Some potential uses of the material are summarized:

| *Application* | *Comment* |
|---|---|
| Briquet and ore binder | Insolubilization with heat occurs at normal core baking period of 400 to 500°F., giving cores with high strength and water resistance |
| Adhesives | Compounds with fire clay to form linoleum paste |
| Emulsifying agent | Effective emulsifier of asphalt and oils and additives to soaps; also used as dispersant and sticking agent for agricultural sprays |
| Soil additive | Aggregates soil into coarse groups, aiding penetration of water and air and retention of water |
| Extender for other organic chemicals | Suitable for use with phenol formaldehyde |
| Surface active agent | Disperses fine particles in water. Useful in ceramic, cement, gypsum, ore flotation, and oil well drilling industries; also has been used in dyeing wool and paper |
| Sequestering agent | Strongly sequesters ferric iron, cupric copper, and stannous tin |
| Wind erosion preventative | Acts as cement, binding soil particles to form crust, preventing wind damage to newly planted crops; also used as road binder |
| Tanning agent | Gives result comparable to typical lignosulphonate (spruce extract) tanning agent |

The compounds are marketed as both the ammonium salts of lignosulphonic acid and substituted sodium salts.

In making these products, stock from the concentrated storage tank is pumped at 30 p.s.i., to the suction side of a stainless steel, high pressure, triplex pump capable of producing 5000 p.s.i. pressure (*12E*). The pump discharges the 50% liquor into the top of a standard 18-ft. diameter spray drier (*19E*), where it is intimately mixed with incoming hot air. All parts of the drier are made of stainless steel, types 304 and 316. The air for drying is heated by a standard direct-fired oil burner which supplies air at 300 to 320°F. (*15E*).

The liquor is dried in suspension, the dried product (containing 4% moisture) falling to the bottom of the conical section of the drier, from where it is conveyed pneumatically through a collecting cyclone and to the dry storage hopper (50 tons capacity) above the packing plant. Here it is filled by an automatic bagger (*22E*) into 50 or 100-lb. valve-type, multiwall paper bags.

The wet gases from the drier with some dried particles pass from the top of the drier to a wet collector where the solids are scrubbed from the gas stream by liquor which is fed into the evaporators. Not only does the wet collector serve as a scrubber, but the heat in the gases is utilized to concentrate the feed liquor prior to evaporation. Gas and rejects from the cyclone separator are also pumped to the wet collector.

In the sodium salts of lignosulphonic acids, the ammonia ion is replaced with sodium. In making these products, caustic soda (50% solution) is added to the evaporation plant feed. The balance of the processing is the same.

The authors wish to acknowledge the assistance of Loren A. La Fond, sulphite superintendent, and R. D. Waddell, technical superintendent, of the Lebanon plant in preparation of this article.

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## PROCESSING EQUIPMENT

1E. Allis-Chalmers Mfg. Co., New York 7, N. Y., vibrating knotter.
2E. Bauer Bros. Co., Springfield, Ohio, pulp cleaners.
3E. Bristol Co., Waterbury 20, Conn., liquid level controller.
4E. Cheney Bigelow Wire Works, Springfield 3, Mass., thickener.
5E. De Zurik Shower Co., Sartell, Minn., consistency regulator.
6E. Electric Steel Foundry Co., Portland, Ore., ESCO cooler.
7E. General American Transportation Corp., Chicago 19, Ill., Conkey flat plate heating surface evaporators.
8E. Glens Falls Machine Works, Glens Falls, N. Y., rotary sulphur burner.
9E. Hesse-Ersted Mfg. Co., Portland, Ore., screening refiner.
10E. Jones & Sons Co., E. D., Pittsfield, Mass., beater.
11E. Jones & Sons Co., E. D., Fibremaster.
12E. Manton Gaulin Co., Inc., Everett, Mass., three-cylinder reciprocating pump.
13E. Morden Machines Co., Portland, Ore., stockmaker.
14E. Nash Engineering Co., South Norwalk, Conn., Hytor vacuum pump.
15E. Peabody Engineering Corp., New York 19, N. Y., direct-fired air heater.
16E. Smith Valley Iron Works Co., Portland, Ore., wood chipper.
17E. Smith & Winchester, Windham, Conn., straight four-drinier paper machine.

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22E. Vredenburg Development Co., Emeryville, Calif., automatic bagger.
23E. Zenith Products Co., West Newton, Mass., positive displacement pump.

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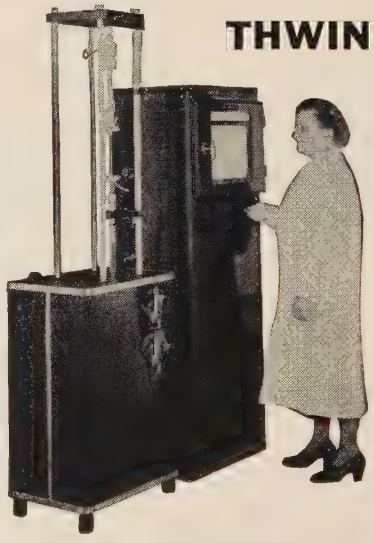

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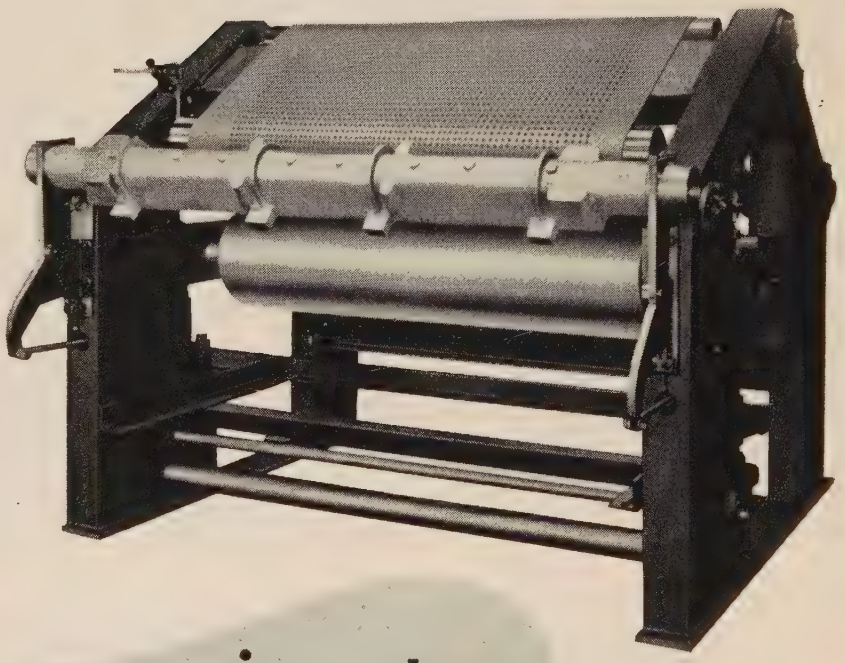
Egan



# air coater

Featuring new advanced design,
the Air Coater coats one side of a web with the
coating metered by a "knife" of high velocity
air. Ideal for photographic, book, label, litho
and adhesive coatings; and sensitized emulsions.

*Send for pamphlets on coating, treating and laminating*

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**Bound Brook, New Jersey**

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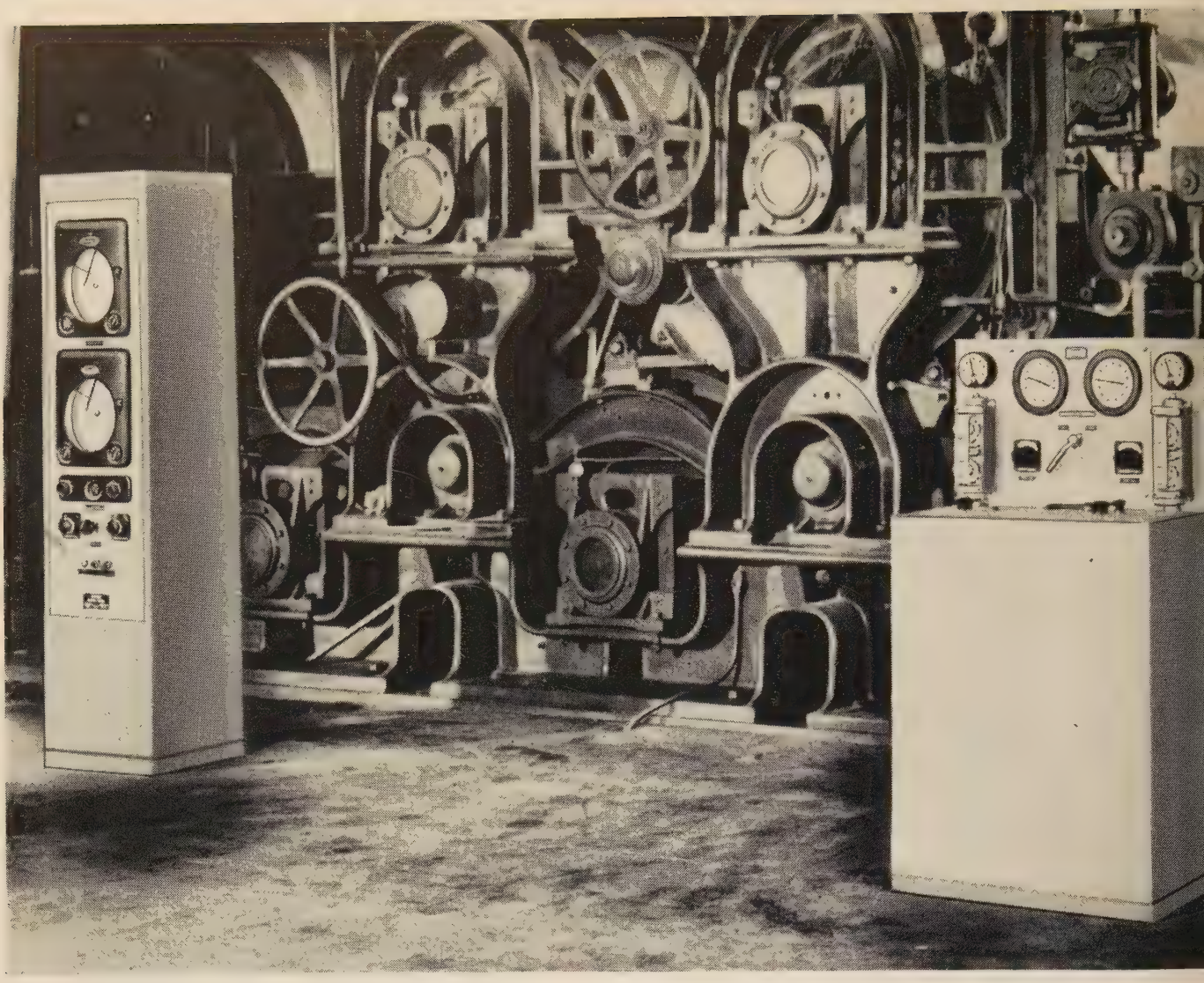


# Increases Tonnage 10% with New

A mill that manufactures dissolving pulp reports, "We've increased our pulp tonnage by 10% since installing a Masoneilan Moisture Control System. And we've cut rejects due to incorrect moisture from 10% to 1%." Another mill manufacturing board claims, "Our production is up 7% a day with Masoneilan's New Moisture Control System."

And that's just part of the story. Typical of many other paper manufacturers of all kinds using Masoneilan Controls, these mills are now making a *better*, *more uniform* paper at *lower cost* with the versatile new Masoneilan Moisture Control System. Here's how and why:

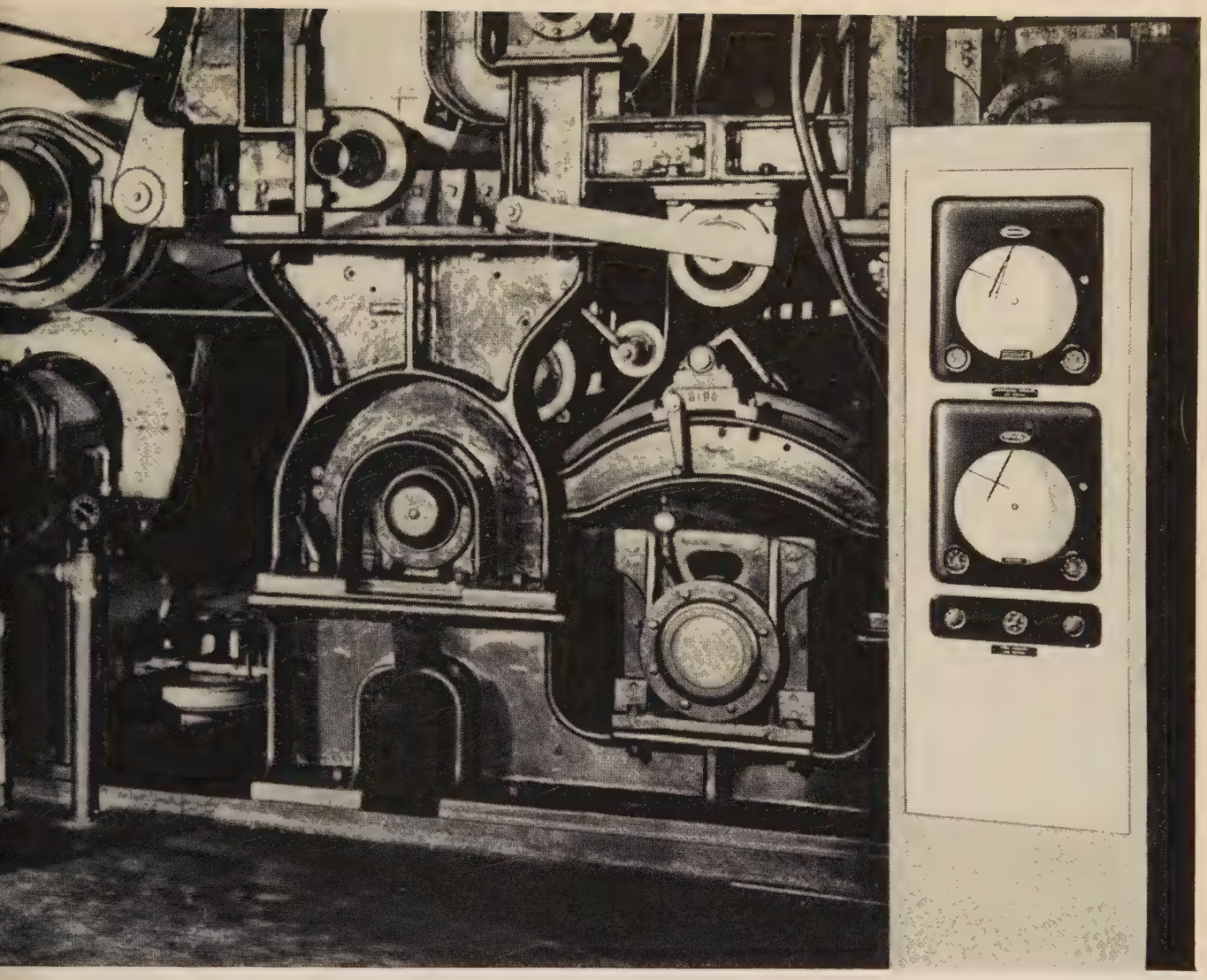
**A Masoneilan Moisture Control System** maintains an average moisture content across the sheet. Does not rely on "spot" measurements.

**It controls moisture content** at each of several points on the machine — from as high as 50% to as low as 3%.

**Sensitive yet stable,** it hold moisture content accurate with ±½ of 1%.

**Can be used for any spee machine** — any width machin Is suitable for *all* grades weights of paper.

**Provides individual pne matic break control** at ea control station. Cuts downtir due to dry breaks . . . speeds covery since it automatica handles steam to machine und all conditions.



# Masoneilan Moisture Control System

**iminates human errors** — akes for more uniform sizing d coating. Pays for itself in a ry short time — in some cases less than a week.

**oints up machine troubles** ch as improper drainage, var- ions in sheet finish, changes in press operation, freeness, consistency, felts, or air removal.

**If you're not presently using it,** you'll want to know more about what this versatile Moisture Control System can do for your mill. Drop us a line and we'll send complete information.

**CONTROLS MOISTURE CONTENT AT . . .**

Breaker Roll

Single or Multiple Dryer Sections

Size Tub

Coating Press

Reel

Masoneilan Moisture Control System is manufactured under Mason-Neilan Patent No. 2,659,987 Dated November 24, 1953.

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**ASON-NEILAN REGULATOR CO.**

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*s Offices or Distributors in the Following Cities:* New York • Syracuse • Chicago • St. Louis • Tulsa adelphia • Houston • Pittsburgh • Atlanta • Cleveland • Cincinnati • Detroit • San Francisco se • Louisville • Salt Lake City • El Paso • Albuquerque • Odessa • Charlotte • Los Angeles Corpus Christi • Denver • Appleton • Birmingham • New Orleans • Dallas • Seattle Mason-Neilan Regulator Co., Ltd., Montreal and Toronto

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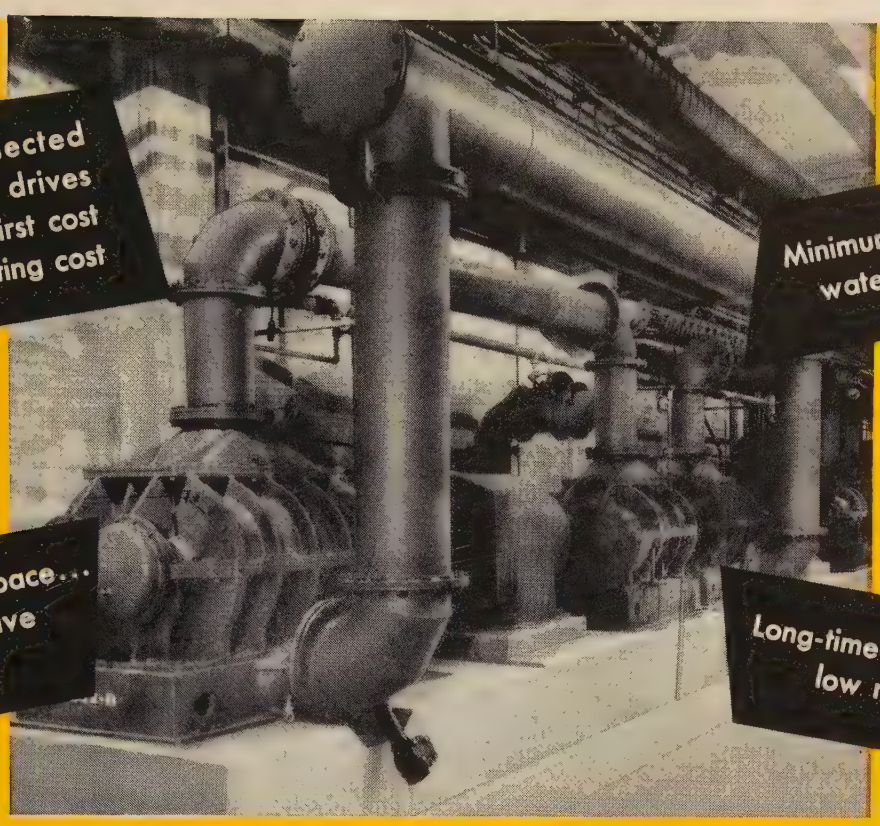
Direct-connected high speed drives to reduce first cost and operating cost
Minimum sealing water needed
Small floor space... less expensive foundations
Long-time reliability... low maintenance

# BE SURE ABOUT THE "BIG 4" *when you select Vacuum Pumps*

To a large extent, these 4 basic values delivered by Roots-Connersville Vacuum Pumps account for the continuing upsurge in R-C installations for paper mills. We can show you figures from users that prove the economies in first cost, and in power and water consumption, because of high-speed (600 rpm or more), direct-connected drives.

Similarly, the records show remarkable freedom from maintenance, with resultant cost reduction, and more important, freedom from down time that can cause serious losses of production. These advantages accrue whether the installation consists of single or compound units—and regardless of capacity. Naturally, the larger the units, the greater the savings, both dollar-wise and percentage-wise.

We suggest that if you are considering vacuum pumps for new installations or replacements, you check with R-C engineers to see how you can reduce your costs without sacrificing any element of long-time, reliable performance. Or, ask for Bulletin 50-B-13 which gives detailed descriptions.

ROOTS-CONNERSVILLE BLOWER · CENTENNIAL · 1854 1954 · A DIVISION OF DRESSER INDUSTRIES, INC.

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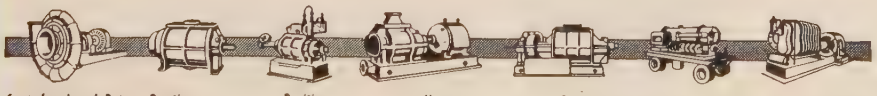

Centrifugal and Rotary Positive Blowers and Exhausters • Positive Displacement Meters • Vacuum Pumps • Gas Pumps • Inert Gas Generators • Spiraxial Compressors

# Paper-making chemicals with "*Service Dividends*"

Many users of SOLVAY's paper-making chemicals consider the practical assistance they receive from the Paper Section of SOLVAY's Technical Service a "Service Dividend."

The Paper Section of SOLVAY's Technical Service can offer services not ordinarily available because:

- These chemists and engineers are especially trained in paper-making and work exclusively with the Paper Industry.
- They have the added knowledge and experience that comes from many years of working solely with the Paper Industry.
- They have at their disposal extensive laboratory facilities and equipment devoted entirely to paper-making and testing, in addition to the general organization and equipment of the world's largest alkali research laboratories.

Your specification of SOLVAY paper-making chemicals will make these "Service Dividends" available to you.

**SOLVAY PROCESS DIVISION**
ALLIED CHEMICAL & DYE CORPORATION
**61 Broadway, New York 6, N. Y.**

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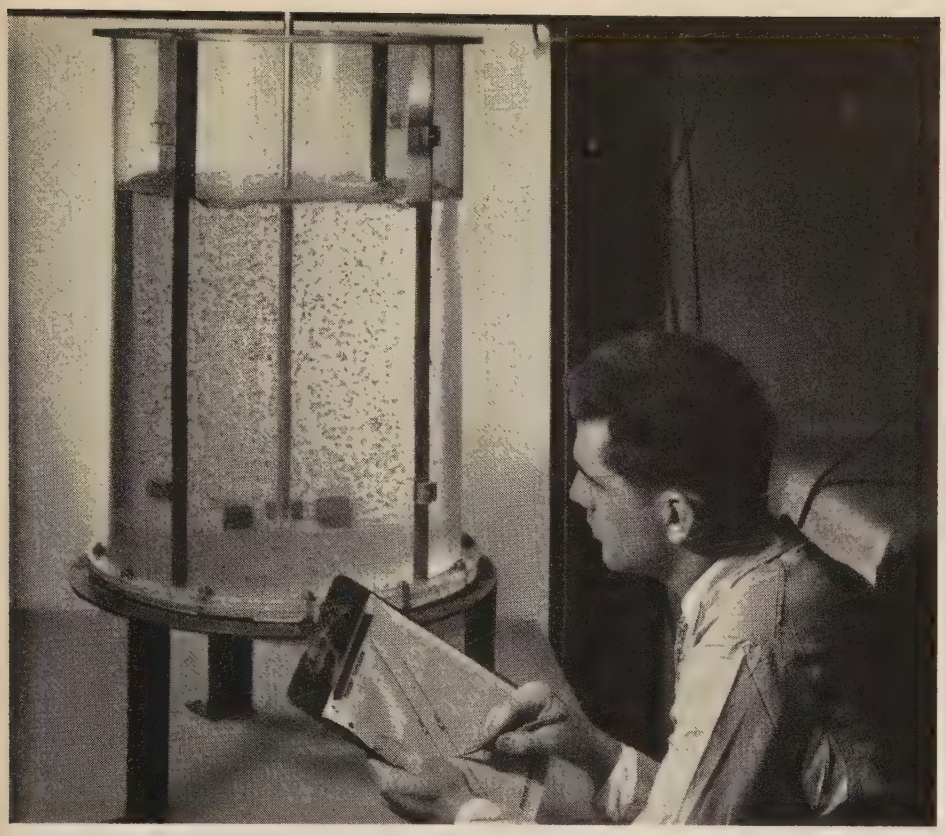
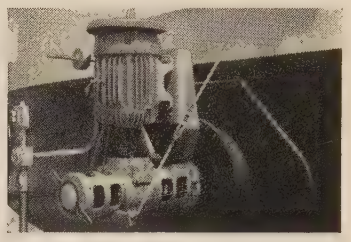
Methyl Chloride • Carbon Tetrachloride



**GET UNIFORM SUSPENSIONS** rapidly, in any size tank, with turbine-type LIGHTNIN Mixers like these. They mix efficiently, and cut maintenance cost because their standard components are easily replaceable from stock. Hundreds of power-speed combinations. Sizes to 500 HP.



**YOU CAN QUICKLY REPACK** a LIGHTNIN Side Entering Mixer (or renew the mechanical shaft seal) without ever draining the tank or dismantling the mixer. New refinements give you lower maintenance cost than ever on rectangular chests and other large tanks. Sizes 1 to 25 HP.

**MIX COATINGS,** sizes, tint solutions rapidly, at low cost, with LIGHTNIN Portable Mixers. Used on any open tank, they need no baffles, and produce uniform suspensions by tumbling and rotating the batch thoroughly. Thousands in use. Thirty models, sizes ⅛ to 3 HP.

# How to keep results constant when you mix fluids

You see here a preview of mixing that can save you money in your coating tanks, starch tanks, or stock chests.

The turbine impeller is working at top efficiency—using minimum power to produce a uniform suspension in least possible time.

It is turning just fast enough to set up the turbulence that picks solids off the tank bottom and sets them in motion.

The impeller is just large enough to set up an over-all fluid flow, moving the materials through and through the tank, with rapid top-to-bottom interchange.

The slightest deviation from 100% mixing efficiency shows up at once on the curve plotted by the Mixco engineer. Using data from thousands of pilot plant runs, he can scale-up this solids suspension to a tank holding five hundred, five thousand, five *million* gallons—quickly, with pinpoint accuracy. Results stay constant—and are fully predictable.

**What's the advantage** of knowing beforehand what sort of mixing results you'll get? Trial-and-error is eliminated. Risk disappears. You may save as much as 40% on initial mixer cost, by eliminating the need for excessive horsepower.

It's easy to get these advantages—and you can learn all about them now without the slightest obligation. Just call in your LIGHTNIN Mixer representative (his name is in your office copy of Thomas' Register). Or write us today.

## Lightnin® Mixers

***GET THESE HELPFUL FACTS ON MIXING***

LIGHTNIN Catalogs contain practical data on impeller selection; sizing; best type of vessel; installation and operating hints; full description of LIGHTNIN Mixers. Yours without obligation. Check and mail coupon today.

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**MIXING EQUIPMENT Co., Inc.**
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# NEW

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# PACKAGING

Huyck papermakers' felts are baled in kraft paper and heavy burlap, assuring clean, safe delivery.

Textile compressive shrinkage blankets are shipped in sturdy, easily stored fibre board drums. The use of a paper product container for shipping felt is a recent development and another *Huyck First.*

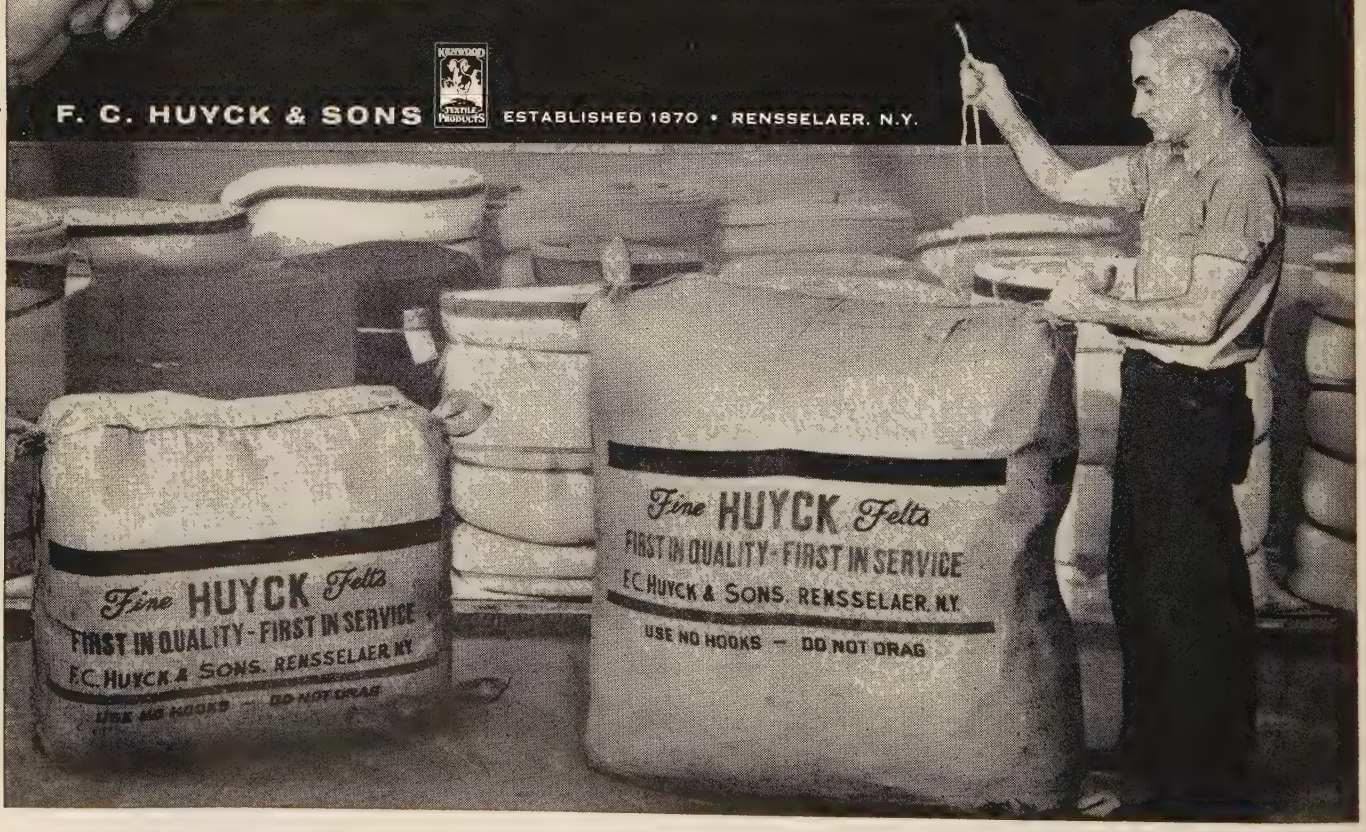
Now, as a further convenience, each Huyck package is clearly labeled in color, for immediate identification, to keep your stand-by Huyck felts ready for instant use.

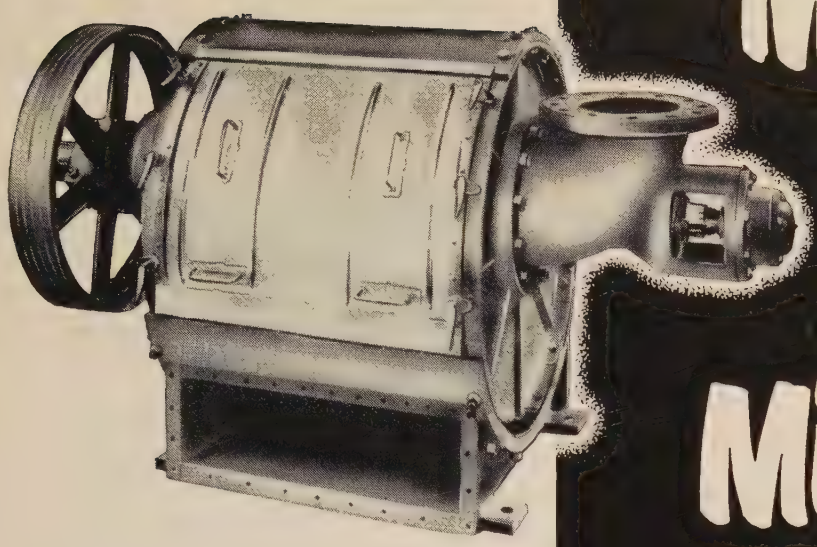
**HUYCK FELTS**
*first in quality...*
*first in service*

*Consider service carefully when you buy felts: it must always be complete and dependable. As the Huyck service record proves, Huyck felts —with Huyck service—mean better production and lower costs for you.*

**Now being issued—Vol. II HUYCK FELT BULLETINS. Where shall we send your copies?**

**F. C. HUYCK & SONS** ESTABLISHED 1870 • RENSSELAER, N.Y.





The "Junior" Screen . . . smallest of *three* units producing from 35 to 150 tons per day, respectively.

# Money Saving Midgets

## Cowan Centrifugal Pulp Screens by

# APPLETON MACHINE COMPANY

Added efficiency, greater economy are the watchwords for *Appleton Machine Company's* junior versions of the standard Mark "A" Cowan Centrifugal Pulp Screen, acknowledged as outstanding in its field.

The Mark "E" Screen is a half-sized model of the standard Mark "A", conservatively rated at a capacity of 2400 U.S.G.P.M. accepted stock. 50 h.p. is required to operate the Mark "E", but its drive is designed to accommodate a 60 h.p. motor, wherever needed. The Mark "E" is particularly advantageous in smaller mills, or as a supplementary screening unit. Also, installing two Mark "E" Screens—instead of a single larger machine—provides a definite safety factor in case of breakdown.

The "Junior" Screen is a quarter-sized model of the big Mark "A", with a rated capacity of 1400 U.S.G.P.M. accepted stock. 25 h.p. operates the "Junior" Screen, but it will handle motors up to 40 h.p. Greatest applications are as secondary screening units, and as a primary screen for mills producing a variety of pulp grades which require a system made up of small, separate units.

Performance of these two Cowan Screens is comparable in every way to that of the standard Mark "A" Screen . . . the same high consistency screening . . . low percent rejects . . . good fiber separation . . . low shower dilution pressure . . . top hydraulic efficiency. Typically sound *Appleton Machine* construction plus a protective coating tailored to fit your needs *complete* your assurance of long-time satisfaction.

CUSTOM-BUILDERS OF PULP & PAPER MILL MACHINES
WINDERS • FINISHING ROLLS • REWINDERS

# CUT YOUR BILLS TO

## YOU GET MORE FOR YOUR DOLLAR WHEN YOU ORDER A SHIPMENT OF PEXOL FORTIFIED SIZE

There are many good reasons why Pexol outsells any other fortified size on the market. Not the least of these is Hercules' unequaled service facilities, which provide every Pexol user with the advantages of a product tailored to his needs. Hercules' technical representatives, trained for service to the paper making industry *exclusively*, are available to visit your plant, study your operating conditions, and recommend a Pexol formulation that will give optimum solubility, overall sizing efficiency and low foam index. Such service is a Hercules "plus"—insures the most for your sizing dollar *in actual use!*

### FACTS ABOUT FOAM

There has been much comment about foam caused by rosin size. The simple fact is: **NO OTHER FORTIFIED SIZE HAS A LOWER FOAM INDEX THAN HERCULES PEXOL**, and at an equal degree of fortification no other size can even approach it.

### RESEARCH FACILITIES

The finest research facilities possible, devoted exclusively to paper making chemicals and their application, are available in helping you improve your product or processing methods. Trained technicians in the laboratory back up Hercules' technical representatives in the field—put at your disposal over forty years of experience in solving paper making problems.

### VARIETY

No one size, dry or paste, can possibly fit every requirement. That's why Pexol is available in both paste and dry forms, and in numerous types. Or, if your needs are unusual, Hercules will develop special modifications of existing formulations to meet specific conditions.

### ECONOMY

Pexol brings reductions in size furnish . . . often as high as 30 to 50%. That means lower inventories, reduced freight and handling cost. Yet sizing tests are maintained . . . often improved. In every way, Pexol is a balanced fortified size that gives the most of all desirable properties—the most for your sizing dollar!

### RESULTS

The final test of any size is how well it works for *you*. Pexol outsells all other fortified sizes. To maintain such a record, it *must* give results! If you're among the few who haven't yet tried Pexol, contact your Hercules technical representative. He'll be glad to demonstrate what you've been missing.

*Paper Makers Chemical Department*

*HERCULES POWDER COMPANY*

INCORPORATED

*967 King St., Wilmington 99, Delaware*

**FOR OPTIMUM SIZING EFFICIENCY, SOLUBILITY, AND LOW FOAM INDEX .**

# SIZE WITH PEXOL

HERCULES®

# PEXOL®

**FORTIFIED SIZE**

PF54-3

# How Celite speeds board output up to 10%

## ...yet improves uniformity and smoothness!

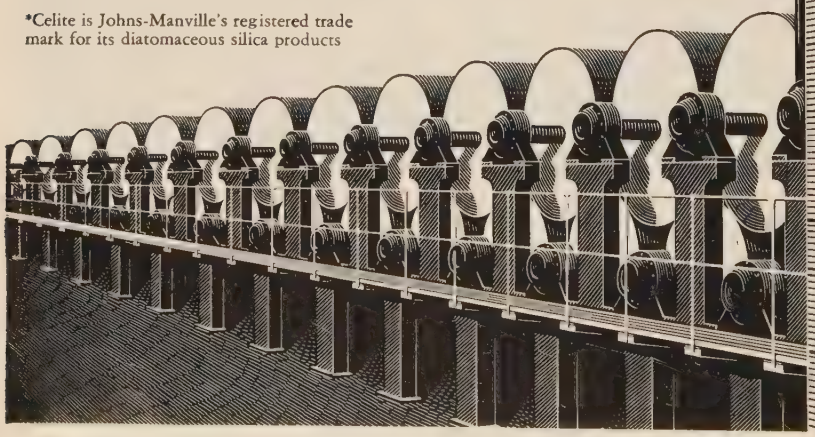
LEADING BOARD MILLS have increased output up to 10% by adding Celite* diatomite powders to their furnish. At the same time they have obtained better quality board with improved uniformity and smoothness. A test run in your mill will quickly demonstrate how you can secure similar results.

You will find that Celite is a highly porous material, with exceptional free-draining characteristics. Since it does not cake up or shrink, it materially improves drainage and drying, makes increased machine speeds possible. And because of its good suspension properties, Celite helps keep fibers evenly dispersed in water, resulting in greater uniformity of finished board.

These unique properties of Celite also permit greater bulk in machine boards such as chipboard, newsliner and solid manila. And for many types of sheet, Celite adds other desirable qualities such as greater opacity, higher brightness at lower cost.

To prove these advantages of Celite, a Johns-Manville Celite Engineer will gladly assist you in making trial runs in your mill. There is no charge for his services. Simply write Johns-Manville, Box 60, New York 16, N. Y.

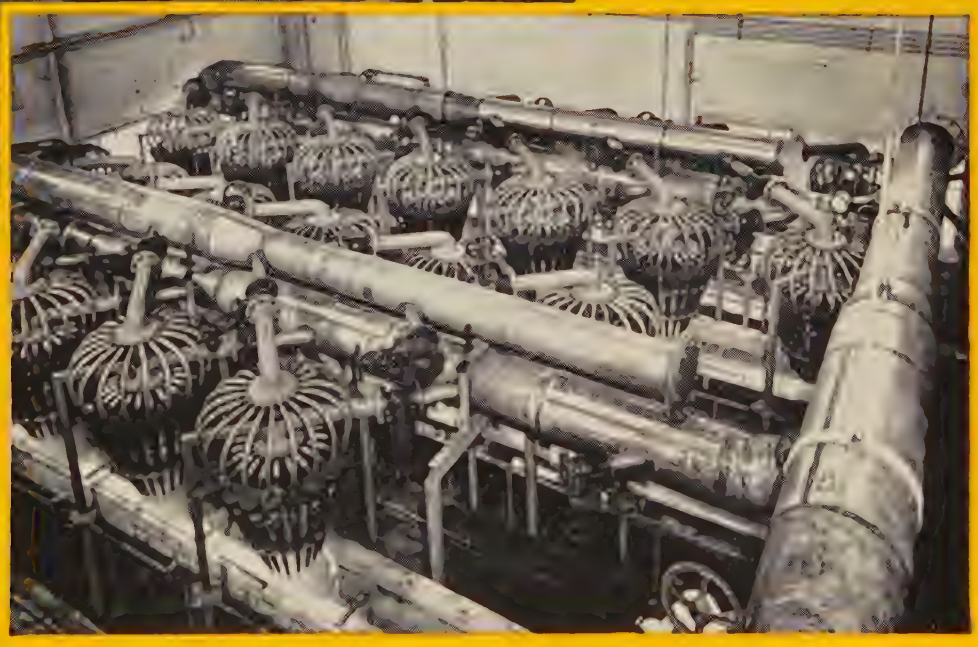
*Celite is Johns-Manville's registered trade mark for its diatomaceous silica products



JOHNS-MANVILLE JM PRODUCTS **Johns-Manville CELITE** PRODUCTS FOR THE PAPER INDUSTRY

## GUARANTEED QUALITY OF SEMI-CHEMICAL PULP *with BAUER REFINERS plus BAUER CLEANERS*

Bauer Double
Revolving Disc Refiner
in a Southern Paper Mill



Clusters of
No. 600 Bauer Cleaners
in a Canadian Pulp Plant

### *A COMBINATION YOU CAN'T BEAT!*

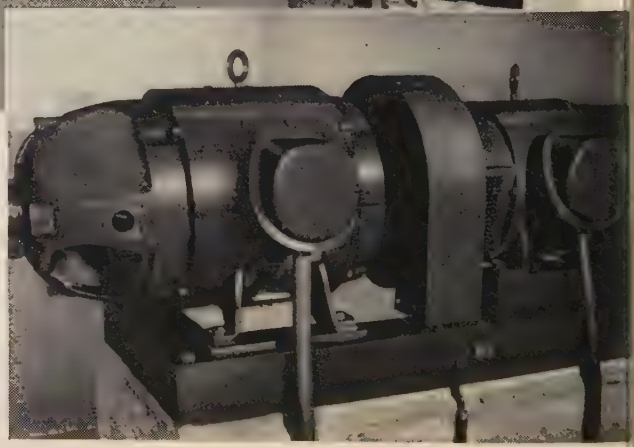
We have the experience and the understanding, as well as the equipment to design your complete pulp processing plant.

*THE BAUER BROS. CO. • 1715 Sheridan Ave. • Springfield, Ohio*

Bauer

**Magamp control** used with differential regulator maintains the proper speed on each section under practically all conditions. It does this regardless of changes in load, variations in vacuum, changes in nip pressure and doctor blade pressures.

**D-c motor** coupled to reduction gear unit—here's a typical section driving unit. Totally-enclosed and force-ventilated, the unit is, in turn, coupled to the paper machine's press section.



**Master frequency m-g set** supplies an adjustable frequency reference to the individual section speed regulators for securing any operating speed. Flywheel compensates for inertia differences.

# Westinghouse drives with Magamp boosted our paper production

Paper machines vary in size, type and number of sections, operating speed range, width of sheet and type of paper produced. In every case, the drive must be engineered to meet these variable factors. After asking you, the papermaker, what you needed most in a drive, Westinghouse modernized its sectional motor drive to give you minimum down time, peak production, and less maintenance.

**Westinghouse Magamp** (Magnetic Amplifier) regulator—a unit that precisely controls section speeds and draws—permits high operating speeds without paper breaks. It's a static device with no tubes and no moving parts other than the cone pulleys and reference motor. Magamp is more rugged, reliable, and requires less maintenance than any other type of regulator now on the market. Elimination of line shafting, with its associated belts, pulleys and clutches, is a safety factor and a space saver. Magamp regulated drive cuts outage time and operating costs. Initial base adjustments, once made, remain unchanged during the equipment's life.

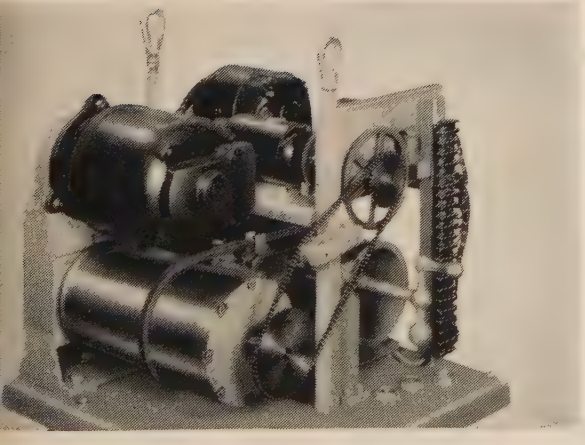
**Every Westinghouse** paper machine drive is an engineered application. Each unit of apparatus is selected to meet job requirements and specific mill conditions. The heavy, rolled steel frame of the Type SK d-c motor, with uniform magnetic qualities, makes it possible to duplicate speed characteristics accurately. Form-wound armature coils, cast brass brushholders, separate field coil units, deep commutator segments, and either sleeve or antifriction bearings provide maximum life and dependability.

**Gear units and gearmotors** are designed and built by Westinghouse to give you dependable and economical power transmission. Motor speeds are reduced to the roll speeds of each driven section by efficient, single helical gears and tapered roller or ball bearings. The exclusive Westinghouse B. P. T. taper-hardened process is used on all gears and pinions. Horizontal split-case construction makes all parts easily accessible for quick, simple inspection without disconnecting couplings, draining the oil, or dismantling the drive.

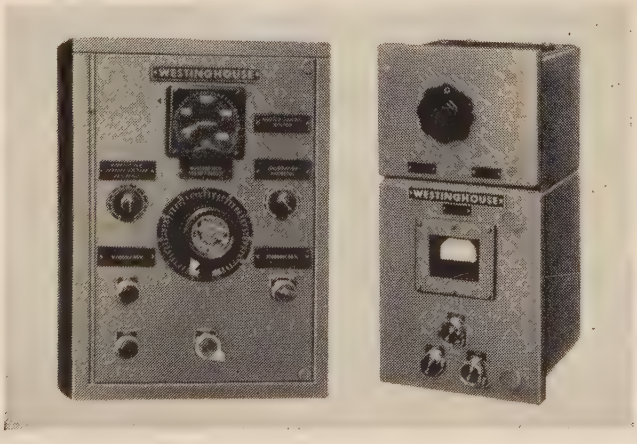
MP-3039

YOU CAN BE SURE...IF IT'S

Westinghouse



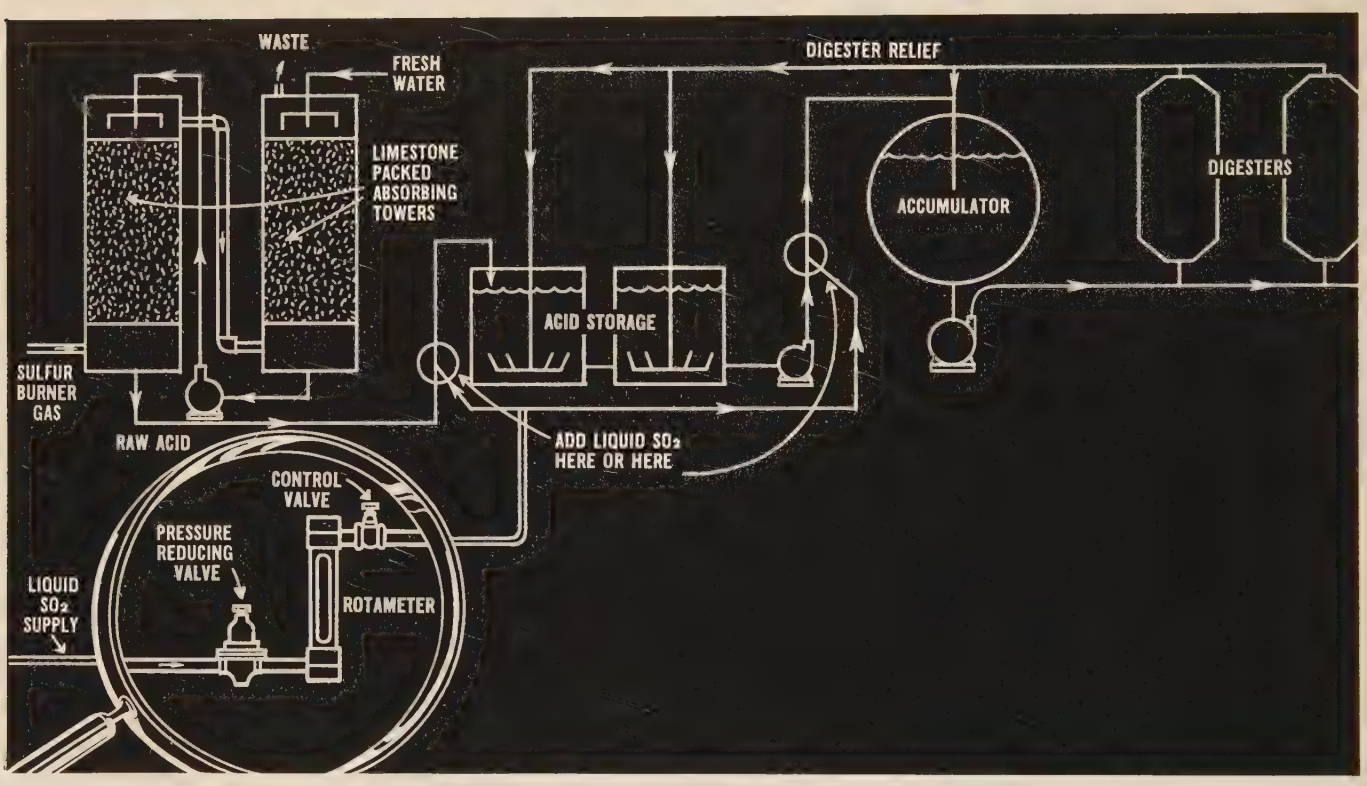
**Error detecting unit** compares the individual section speed with the master set frequency. Any difference is transmitted to the Magamp regulator which brings the section speed in step.




**Operator's master control** station (left) shows speed indicator and provides for automatic control of machine functions. An individual operator's station (right) has a meter to show load.

SULFITE MILL OPERATORS:

# cut cooking time improve quality




with easy-to-operate

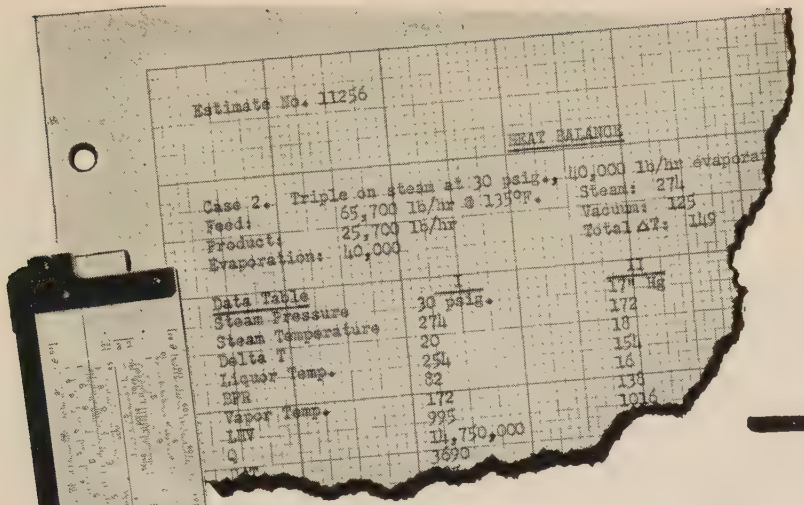
# Ansul sulfur dioxide system

Installing an Ansul sulfur dioxide system is the sure, economical way to cut cooking time and keep sulfite pulp production going at an even rate *in spite of* wet or green wood cooks, high water temperatures or other hard-to-control variables.

Adding free sulfur dioxide to the acid supply gives faster wood chip penetration thereby increasing the rate of pulping. The increased pulping rate gained by adding free sulfur dioxide permits a considerable reduction in the pulping temperature for a constant digestion time. This cuts fuel costs and increases quality because lower temperatures give higher yields and improved chemical and physical properties to the finished product.

Why not further investigate the many advantages to be gained by installing Ansul's simplified sulfur dioxide system. Write today for more information. ANSUL CHEMICAL COMPANY, Department C-45, Marinette, Wisconsin.

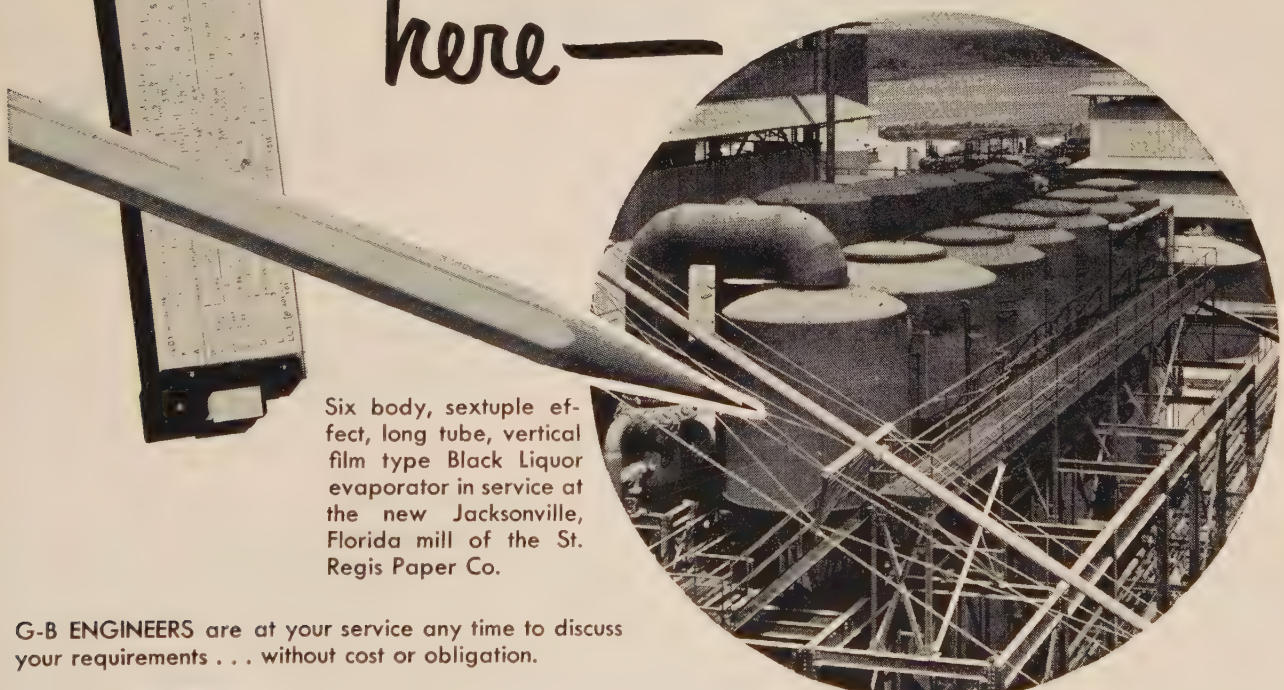
ANSUL


Estimate No. 11256
HEAT BALANCE
Case 2. Triple on steam at 30 psig., 40,000 lb/hr evapora
Feed: 65,700 lb/hr @ 135°F. Steam: 274
Product: 25,700 lb/hr Vacuum: 125
Evaporation: 40,000 Total ΔT: 149
Data Table
Steam Pressure 30 psig. 274
Steam Temperature 274 20
Delta T 20 254
Liquor Temp. 254 82
BPR 172 995
Vapor Temp. 995 11,750,000
LMV 11,750,000 3690
Q 3690

# The right start here

Given only the process conditions, GOSLIN-BIRMINGHAM Engineers are prepared to design chemical process installations for your plant and take *full responsibility* for the process, workmanship and material. This complete responsibility, inside one fence, under one roof and by one organization . . . from slide rule to freight car . . . assures you of the ultimate in maximum efficiency at minimum cost!

## cuts costs here—



Six body, sextuple effect, long tube, vertical film type Black Liquor evaporator in service at the new Jacksonville, Florida mill of the St. Regis Paper Co.

G-B ENGINEERS are at your service any time to discuss your requirements . . . without cost or obligation.

**If you plan to attend the TAPPI Convention Oct. 6-7-8, or the Southeastern Regional Meeting of the A.C.S. . . . both in Birmingham . . . be sure and visit our "hospitality" suite at the Dinkler-Tutwiler Hotel.**

GB

**GOSLIN-BIRMINGHAM MANUFACTURING CO., INC.**

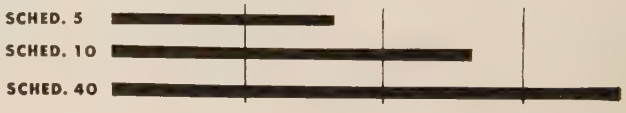
**BIRMINGHAM • ALABAMA**

# when you need STAINLESS PIPE...

## look at all 3




*Here's A Cost Comparison on All Three*




*Here's How All Three Compare in Weight*

To get the most economical stainless pipe for handling corrosive fluids, Schedules 5, 10, and 40 should be closely compared for:

*initial cost*
*installed cost*
*ease of handling*
*flexibility of assembly*
*flow capacity*
*adequate allowable working pressure*
*effective corrosion resistance*
*service life*

Whichever Schedule—5, 10 or 40—is best for your service conditions, Carpenter makes it to meet ASTM pipe specifications. Carpenter Stainless Pipe is available in sizes up to 4 inches, depending on weight . . . in AISI stainless analyses plus Carpenter Stainless No. 20 Cb, and Carpenter Alloys B and C for super corrosion resistance. For services requiring extra smooth surfaces, Carpenter Stainless Pipe can be supplied with 80 to 320-grit polished I.D. and/or O.D. finishes. Adequate stocks are as close as your local distributor. Call him for information and time-saving delivery.

**The Carpenter Steel Company, Alloy Tube Division, Union, N. J.**

*Export Dept.: The Carpenter Steel Co., Port Washington, N. Y.—"CARSTEELCO"*

**Carpenter**

more than corrosion resistance

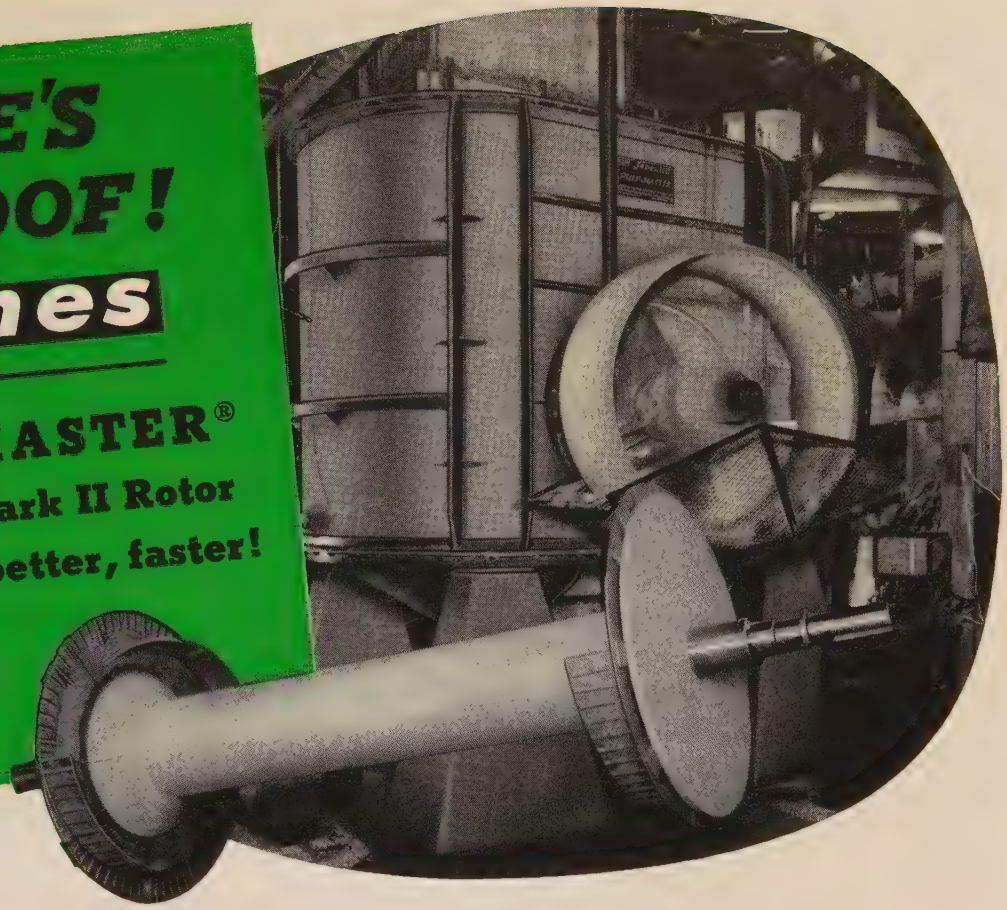
**Stainless Tubing & Pipe**

# HERE'S PROOF!

E.D. Jones

## PULP-MASTER®

**with new Mark II Rotor defiberizes better, faster!**



"The results are as good as any we could wish for," says the production manager of a mill making sulphite and sulphate bonds. "We've reduced the horsepower hours for pulping bond broke from 187 to 60."

*The slides tell the story.* Taken from an actual run, under mill conditions, they graphically demonstrate the superiority of the Pulp-Master with the new Mark II rotor over conventional pulping methods.

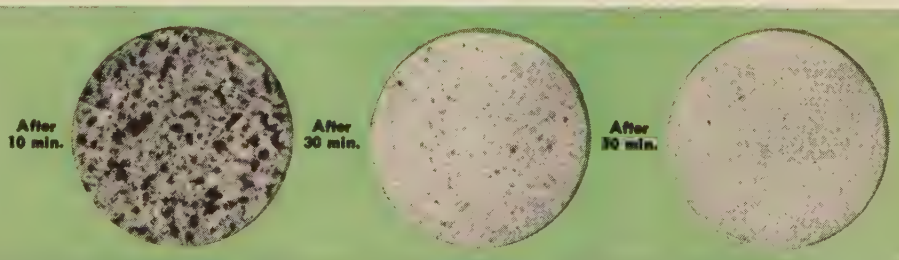
The Pulp-Master is equally effective on baled pulp, broke, bagasse, waste paper or other material. Ask your Jones representative — or write direct for details.

E.D. Jones

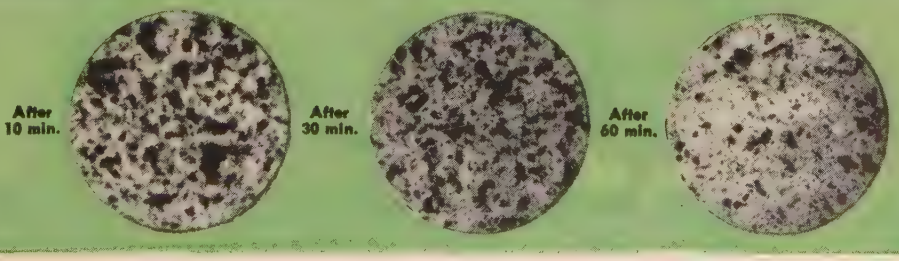
**E. D. JONES & SONS COMPANY**
**Pittsfield, Mass.**

**BUILDERS OF QUALITY STOCK PREPARATION MACHINERY**

**Actual unretouched "shadowgraph" photos of papers made of stock taken from run of hard-pressed unbleached pulp in PULP-MASTER with Mark II Rotor, at intervals shown.**




**Actual unretouched "shadowgraph" photos of papers made under same conditions from same stock while being defiberized by conventional pulping method, at intervals shown.**



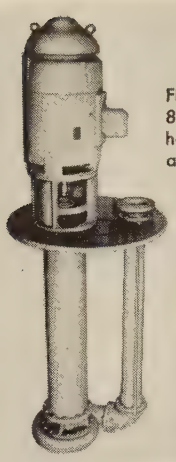


Fig. 279 — Lawrence 8-inch Vertical Pump for handling molten salts at 850°F.

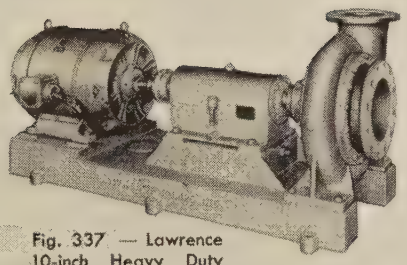


Fig. 337 — Lawrence 10-inch Heavy Duty Stainless Steel Chemical Pump.

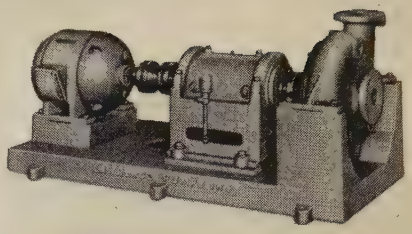


Fig. 343 — Lawrence Steam-Jacketed Chemical Pump with water-cooled bearings.

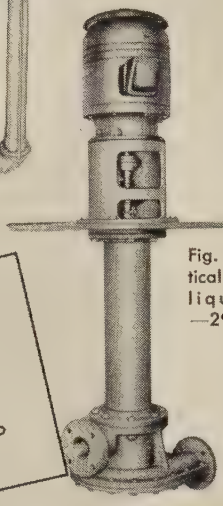


Fig. 307 — 2-inch Vertical Pump for handling liquid oxygen at —297°F.

# SPECIAL HANDLING
## for the process industries

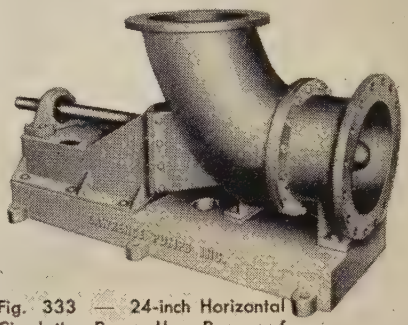
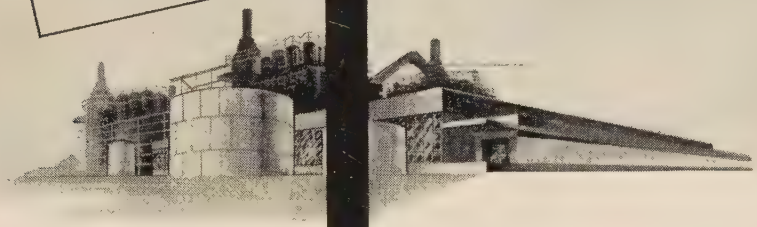


Fig. 333 — 24-inch Horizontal Circulating Propeller Pump of Nickel Alloy Construction.



Send for Bulletin 203-6 for a complete summary of acid and chemical pump data.

## LAWRENCE PUMPS

***handle every fluid that can be pumped - corrosive, abrasive, hot (1500°F) or cold (-347°F)***

To pump liquid oxygen, liquid chlorine, mercury, hot sulphuric acid, molten salts, molten metals, heavy abrasive slurries, etc., requires engineering skill normally not available from a pump manufacturer. Lawrence engineers have specialized in this difficult field for more than 90 years. It may almost be said — there is no "pumpable" fluid with which Lawrence engineers have not had repeated and successful experience.

Lawrence Pumps are designed for either horizontal or vertical operation. Every pump is custom built from the corrosion- and abrasion-resistant metals best-suited to the fluid to be pumped.

If you have a particularly difficult pumping problem, we can save you both time and money. Write us the pertinent details. No obligation.

## LAWRENCE PUMPS, INC.

357 Market Street, Lawrence, Massachusetts

# Control that Pitch!

In the manufacture of fine papers, pitch control is an absolute must. The only question: what is the most effective, most economical way?

For the pitch conditions which prevail in most mills, you'll find that Nopco® 1187-X, a dehydrated naphthalene condensate readily soluble in water, tends to keep your screens, wires, and press rolls free from pitch.

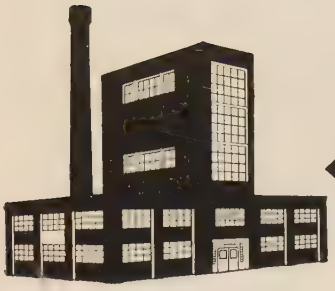
You'll need only from 2 to 4 pounds of Nopco 1187-X per ton of stock. You'll find clogging of your screens and wires will be minimized, your press rolls less liable to stick. Just drop Nopco 1187-X, dry, in the beater before you add alum. Or if preferred, you can dissolve it in water and introduce it at any point befor the screens. Either way, you'll control pitch and speed production.

Write today for testing sample of Nopco 1187-X. Nopco Chemical Company, 444 Water Street, Harrison, N. J.

NOPCO

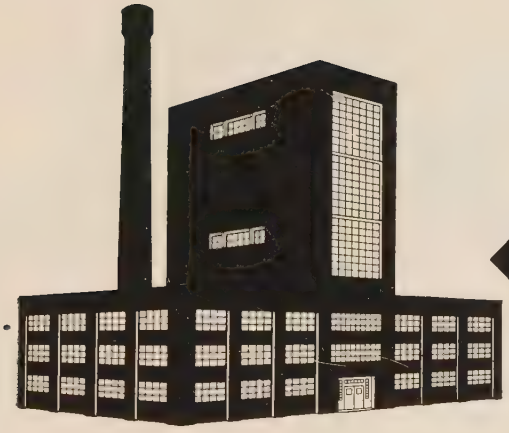
PLANTS: Harrison, N. J. Cedartown, Ga. Richmond, Calif.

# KAMYR
## continuous cooking



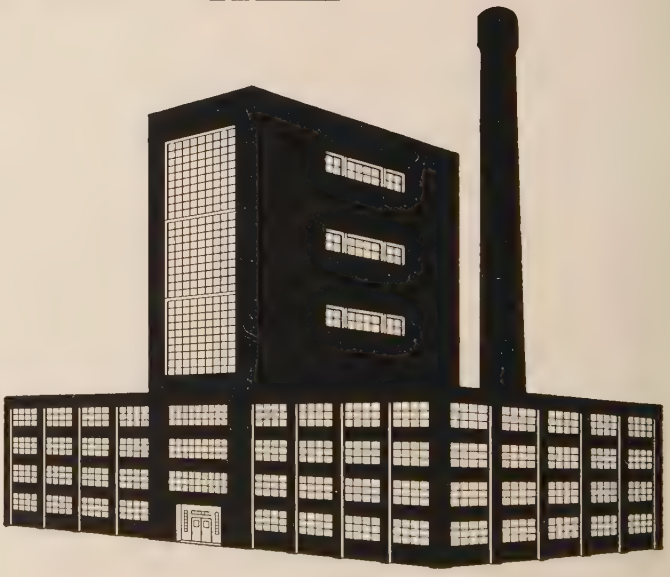
30T/24H

50T/24H

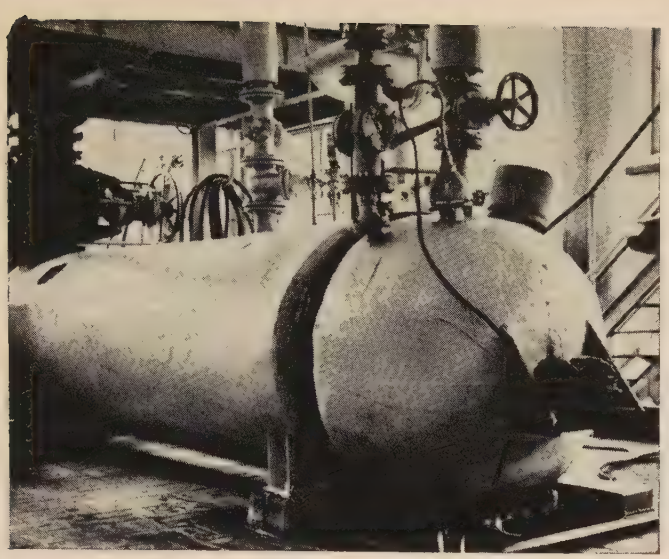


100T/24H

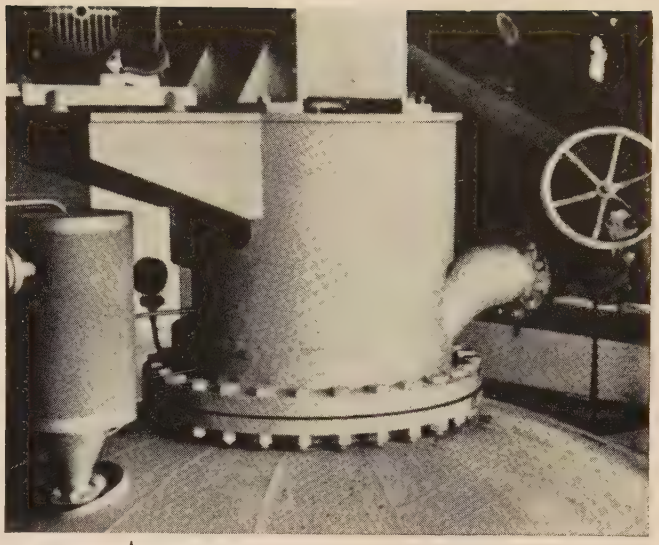
*And Now:*
200T/24H



For Information Contact **KAMYR INC.** Hudson Falls, N. Y.

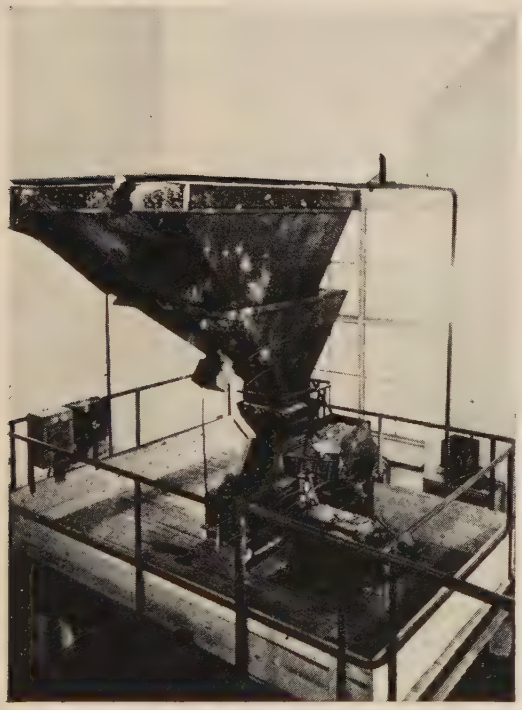


STEAMING VESSEL



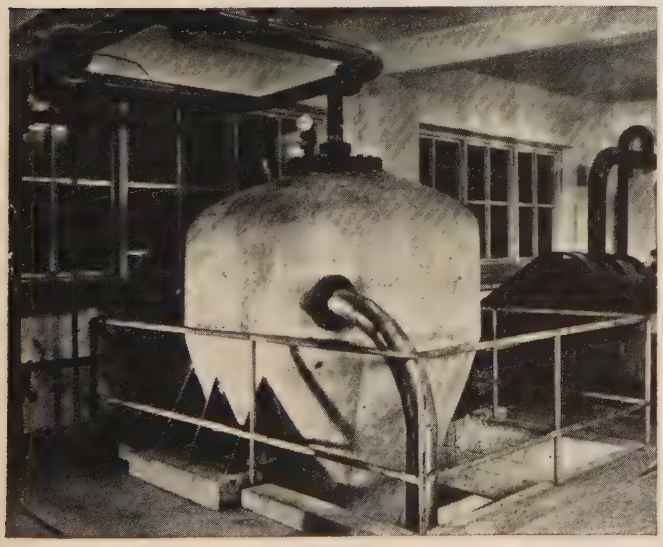
TOP OF DIGESTER

# KAMYR CONTINUOUS COOKING OPERATING VIEWS

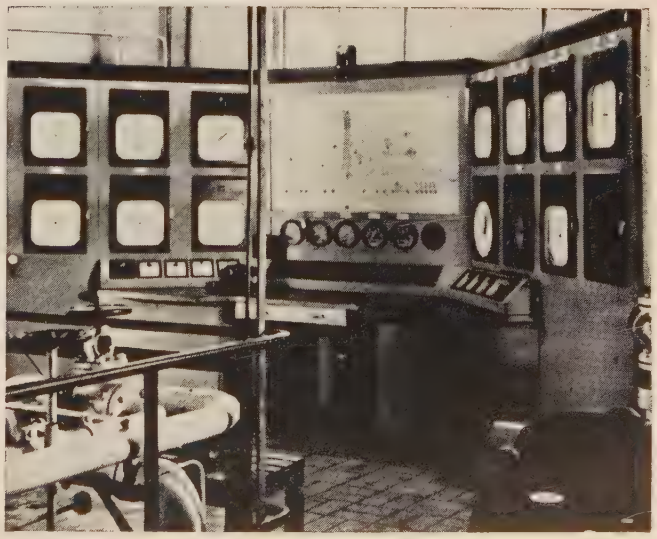


CHIP FEED

TOP OF BLOW TANK



INSTRUMENT PANEL



# AUTOMATIC TENSION CONTROL...

IS ONLY ONE OF MANY EXCLUSIVE FEATURES INCORPORATED IN THE

## S&S SHAFTLESS, PIVOT TYPE MILL ROLL STANDS

THESE double, hanger, all electric shaftless mill roll stands carry two 3-ton rolls simultaneously. Rolls are mounted from the same loading station and moved electrically in an arc to the spending side with the web always under automatic constant tension from 60″ diameter right down to the core.

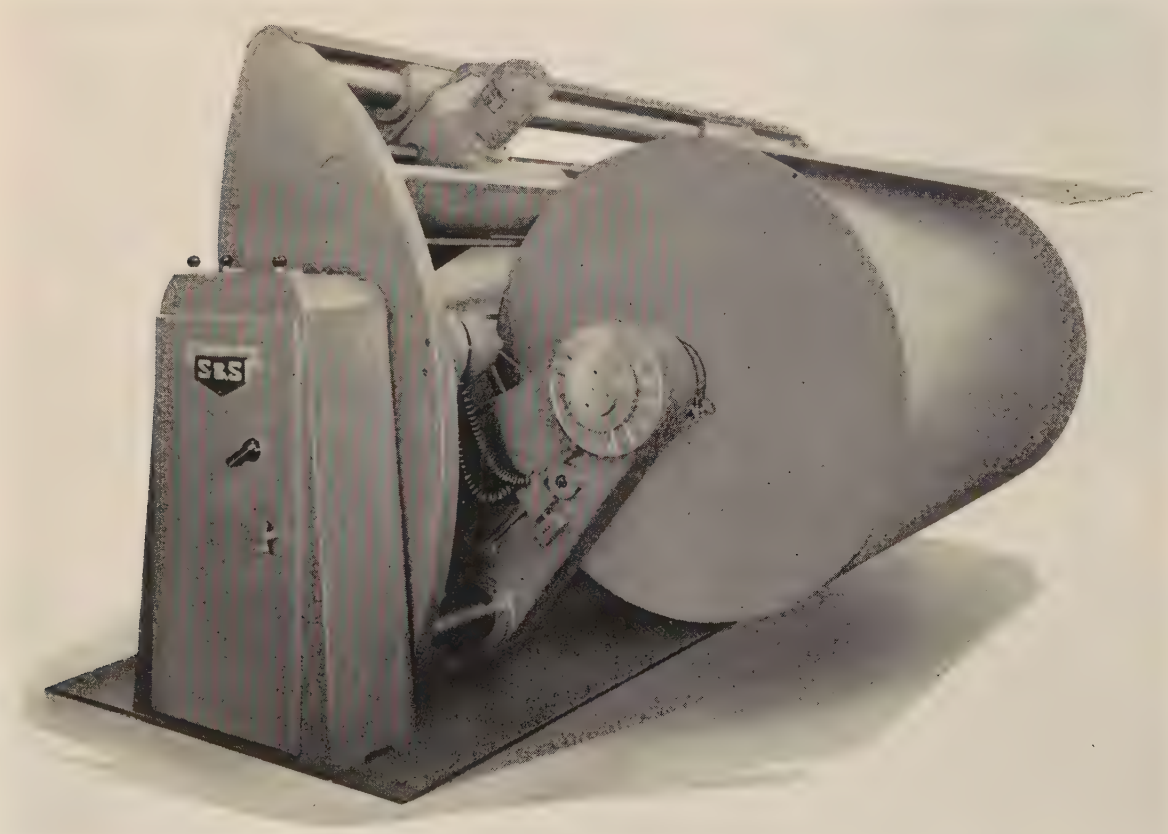
Core plugs are pushed in or out of core holes electrically and also electrically lined up with other rolls.

Designed for any machine requiring feeding of heavy rolls of paper.

Overhead rails and hoists, aisle loading, portable shafts, plugs and brakes entirely eliminated.

No heavy lifting, practically eliminating toil as well as reducing manpower.

Splicing is always done on the outside.



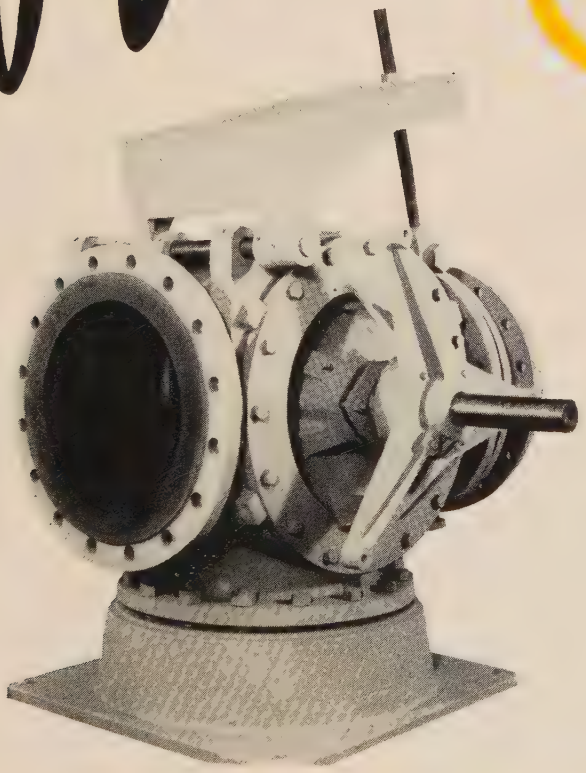

*Established 1917*

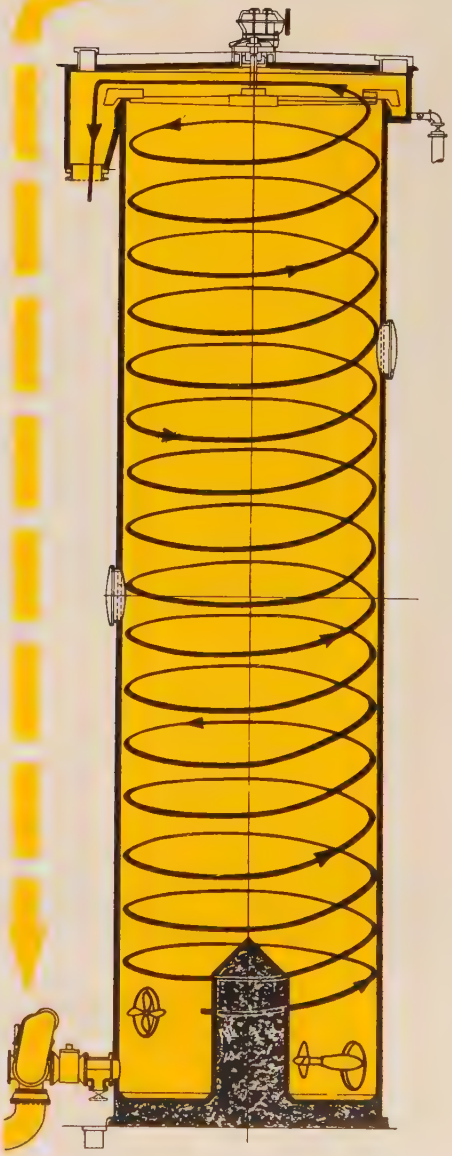
**S & S CORRUGATED PAPER MACHINERY CO., Inc.**

**161 NORTH FOURTH STREET** **BROOKLYN 11, NEW YORK**

# Good Bleaching

## STARTS HERE





## IMPCO CHLORINE MIXER

Of the various stages in the modern bleach plant, the chlorination stage is the most difficult to control from a standpoint of mixing the chlorine *completely* with the pulp.

The IMPCO double wabble plate low density Chlorine Mixer gives a quick intimate mixing result which improves retention in the main tower and insures uniformly chlorinated pulp, with significant savings in chlorine.

**IMPROVED**
**MACHINERY INC.**
**NASHUA, NEW HAMPSHIRE**

*Sherbrooke Machineries Ltd., Sherbrooke, Quebec, manufacture similar equipment in Canada*

# FORMATION

## THAT'S OUR JOB

Few of us here at Valley can remember when we haven't lived and slept the daily problems of designing and building Inlets and Headboxes. Our specialization goes back that far.

And we have followed them into the field, observing and refining every detail of performance . . . . . all resulting in a continuously enlarging reservoir of information to assist our mill customers in the making of *better* paper at *lower* cost.

*Experience* is a great teacher, and we have taken fullest advantage of this practical source of knowledge to serve you.

*Better formation* is *your* objective.

*Better formation* is *our* job.

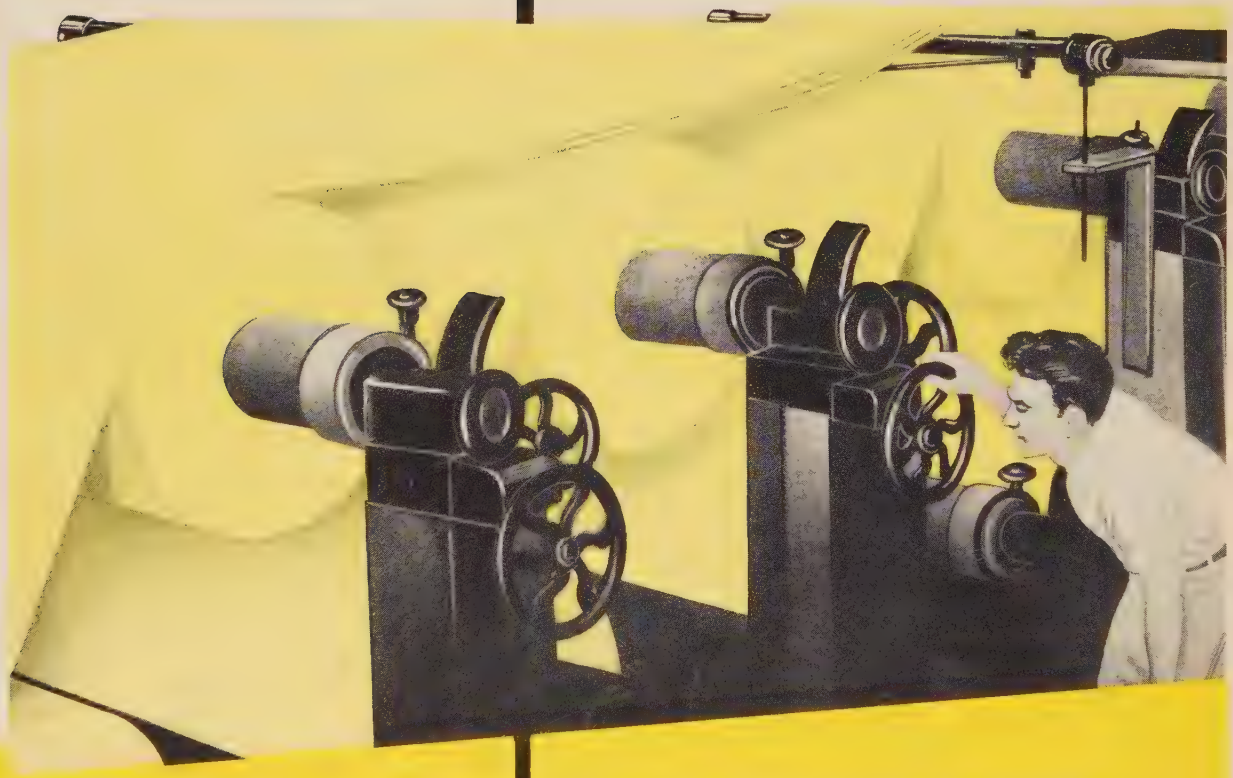
We invite you to submit your formation problems to us for consultation.

*Stock Entrance Equipment by*

**VALLEY**
**Iron Works Company**
**APPLETON, WISCONSIN**

# *How YOU can profit by OUR experience*

Over the years, we have noticed that mills with complete color lines can usually keep paper-making machines operating profitably when adequate tonnage on more competitive lines is hard to get. We have helped many of these mills to get started on colored paper and board specialties.



Running colored lines now involves fewer difficulties than you may suspect. With the proper formulas and methods, they can be quite profitable indeed. From first hand experience, National Aniline paper technicians well understand the aspects of making colored lines. They can provide very practical help on formulas and application methods. This is part of our comprehensive service to paper mills. We invite you to use it.

# NATIONAL PAPER DYES

**NATIONAL ANILINE DIVISION** ALLIED CHEMICAL & DYE CORPORATION 40 Rector Street, New York 6, N. Y.

BOSTON PROVIDENCE PHILADELPHIA CHICAGO SAN FRANCISCO PORTLAND, ORE. GREENSBORO CHARLOTTE RICHMOND ATLANTA COLUMBUS, GA. NEW ORLEANS CHATTANOOGA TORONTO

# *Planning for Posterity...*

A Tree Farm is privately owned land dedicated to continuous growth of forest crops for commercial purposes. The words "Tree Farm" indicate that the owner has agreed to manage his forest lands according to sound forestry principles.

Tree Farming, like other forms of crop raising, is a practical business. The Weyerhaeuser Timber Company established the first certified Tree Farm — the Clemons — in 1941 at Montesano, Washington. Thus began a nation-wide movement toward a continuous production of forest crops among private owners of forest lands.

Weyerhaeuser Timber Company today has eleven Tree Farms under *cultivation.* Adequately protected from fire, insects, disease, and destructive grazing, these Tree Farms insure a constant supply of pulpwood and other forest products.

This Cellulose Age demands an uninterrupted source of raw material. Weyerhaeuser Timber Company has *planned* timber crops — for now, and for the *future,* assuring industry of a long-term never-ending supply of woodpulp.

PULP DIVISION

WEYERHAEUSER

*for economical* SEMI-CHEMICAL *pulping*

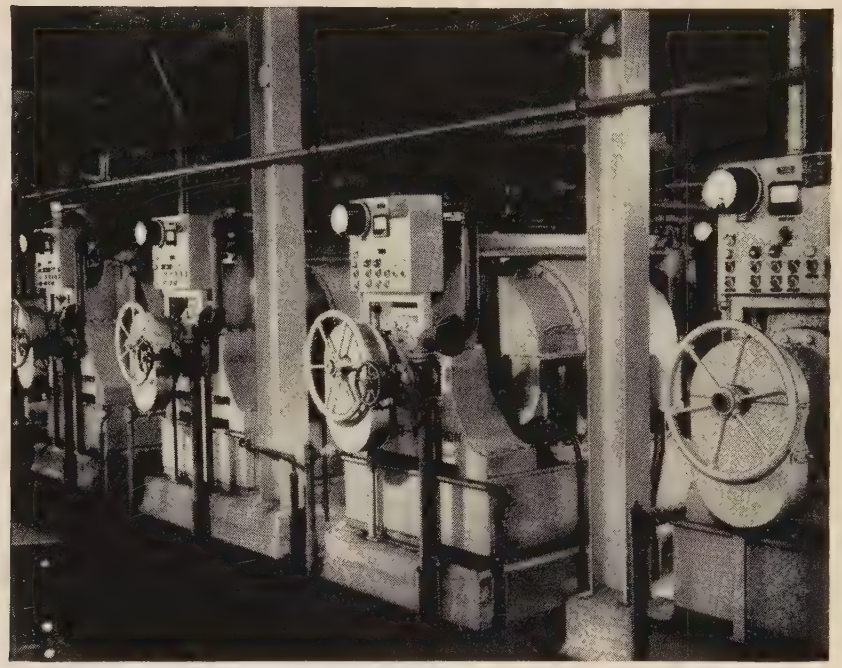
# AMERICAN BOX BOARD COMPANY

## *continually chooses*

## SPROUT-WALDRON

## 36-2 REFINERS

The SPROUT-WALDRON refiner is the leading producer of semi-chemical pulp



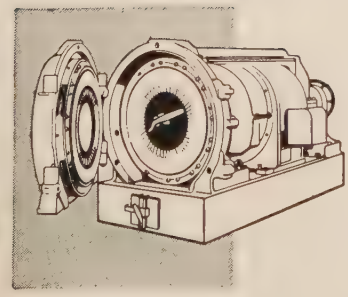
Four of the eight Sprout-Waldron 36-2 refiners at American Box Board Company, Filer City, Michigan. All eight units refine neutral semi-chemical chips for 9 point corrugating board in this most modern semi-chemical mill. Two were installed in 1948, one in 1949, four in 1952 and one in 1954. Repeat orders, such as this, are the best evidence of customers' satisfaction with Sprout-Waldron Refiners.

*Single disc design, peripheral control ring and rugged construction for...*

- **high pulp quality**
- **high capacity**
- **flexibility of operation**
- **low maintenance**

*For more information on semi-chemical pulping, or any other pulping application, send for our file of technical and practical data. Write to Sprout-Waldron & Co., Inc., 38 Logan Street, Muncy, Pa.*

SW



*for your pulping problem—*

**SPROUT-WALDRON**

**PULP REFINERS**

# Progress in Alkaline Pulping—1953*

J. E. STONE

## EXPANSION, MODERNIZATION, NEW MILLS

### *United States*

*Paper Mill News* (*1*) has listed in alphabetical order by companies the new mills or equipment which started up in 1953, are under construction, or are projected for 1954–55. Moon (*2*) outlined the plans for the new newsprint and kraft mill of Bowaters at Calhoun, Tenn., which is expected to start operations in 1954. The initial capacity will be 125,000 tons of newsprint and 50,000 tons of either bleached or unbleached kraft market pulp. Eaton (*3*) described the modernization and expansion of the Halifax Paper Co. mill at Roanoke Rapids, N. C., which was also described in the *Paper Trade Journal* (*4*). *Pulp & Paper* (*5*, *6*) reported on the new 250-ton per day chlorine dioxide–bleached kraft pulp mill of the East Texas Pulp & Paper Co. at Evadale, which is scheduled for start-up in early 1955. Pearson (*7*) and *Pulp & Paper* (*8*) described the 3-year expansion program of the Hollingsworth & Whitney mill at Mobile, Ala., which is now completed. The production capacity for dissolving sulphate pulp at the International Paper Co.'s Southern Kraft Division (Natchez, Miss.) has been doubled (*9*, *10*). Descriptions of Mead Corp.'s 110-ton bleached soda pulp mill at Chillicothe, Ohio, have been given (*11*, *12*). In addition to the mixtures of aspen, willow, cottonwood, yellow poplar, and gum previously used, the mill will also handle oak which is the most abundant species in the area.

The 7-year modernization program of the Mosinee Paper Mills Co. which has led to the present daily production of 165 tons of special sulphate papers has been described by McPherson (*13*). Another Wisconsin kraft mill, that of Nekoosa-Edwards Paper Co. at Nekoosa, has been modernized and expanded to 200 tons per day. Descriptions of the mill and equipment have been given by Nugent (*14*), Kassing (*15*), *Paper Industry* (*16*), and *Pulp & Paper* (*17*). Roth, *et al.* (*18*) described the instrumentation program in all departments of the new sulphate mill of the Riegel Corp. at Riegelwood, N. C. Several descriptions have appeared of the St. Regis mill at Eastport on the outskirts of Jacksonville (*19*, *20*, *21*). This mill started operation on Jan. 14, 1953, and has an annual capacity of 100,000 tons of kraft pulp, paper, and board. A progress report appeared in the *Paper Trade Journal* (*22*) concerning the modernization and expansion program at each of the West Virginia Pulp & Paper Co.'s mills at Charleston, Covington, Luke, Tyrone, Williamsburg, and Mechanicville. Weyerhaeuser's new 250-ton bleached kraft mill at Everett, Wash., is now in full-scale production and a number of descriptions of it (*23*), together with the Longview mill (*24*), have appeared.

*Bagasse.* It is reported (*25*) that the 50-ton per day newsprint mill at Lockport, La., utilizing bagasse, is expected to start operations at the end of 1953.

### *Canada*

There has been an expansion from 130 to 175 tons per day of unbleached kraft at the Port Mellon, B. C., mill of the Howe Sound Pulp Co., Ltd. (*26*). At Harmac, B. C., MacMillan & Bloedel, Ltd. have expanded to 210,000 tons, making the mill the second largest in the world for bleached sulphate. Numerous articles have appeared concerning this expansion (*27–32*), *Canadian Pulp & Paper Industry* (*30*) devoting almost an entire issue to it.

### *South America*

In Brazil a mill for pulping bagasse by the Celdecor-Pomilio process is under construction (*33*).

### *Europe*

A new straw pulp mill at Arnhem, Holland, using the Celdecor-Pomilio process has been described (*34*) as the most modern bleached straw pulp mill in the world. In Sweden, the modernization program at the kraft mill belonging to Billeruds Aktiebolag has raised production to 65,000 tons per year (*35*).

### *South Africa*

The paper industry in South Africa has been discussed (*36*) and a new kraft mill at Mandini described (*37*). It is expected to be ready in early 1954. Production will be 100 tons per day, using pine and a eucalypt for raw material.

### *Pakistan*

A sulphate mill now under construction will utilize bamboo, producing 30,000 tons of pulp annually (*38*).

### *New Zealand*

A new sulphate mill at Kinleith was expected to be in operation by the end of 1953 (*39*, *40*, *41*). Part of the output will be exported to Australia for the newsprint industry. Monterey pine ((*Pinus radiata*) is used for raw material.

## ALKALINE PULPING—GENERAL

The operations and typical equipment of a kraft pulp mill was described by Jones (*42*). Recent developments in sulphite and sulphate pulping were reviewed by Bialkowsky (*43*) who discussed, among other things, the introduction of bleached Douglas-fir kraft market pulp at the Weyerhaeuser Timber Co. A brief description has been given (*44*) of the sulphite and sul-

J. E. Stone, Research Assistant, The Institute of Paper Chemistry, Appleton, Wis.

*Sponsored by the Alkaline Pulping Committee of the Technical Association of the Pulp and Paper Industry, M. B. Pineo, Chairman. The *Southern Pulp and Paper Manufacturer* finances the preparation of this report annually for TAPPI as a sevicce to the pulp, paper, and paperboard manufacturers.

# SCIENTIFIC CONSTRUCTION*

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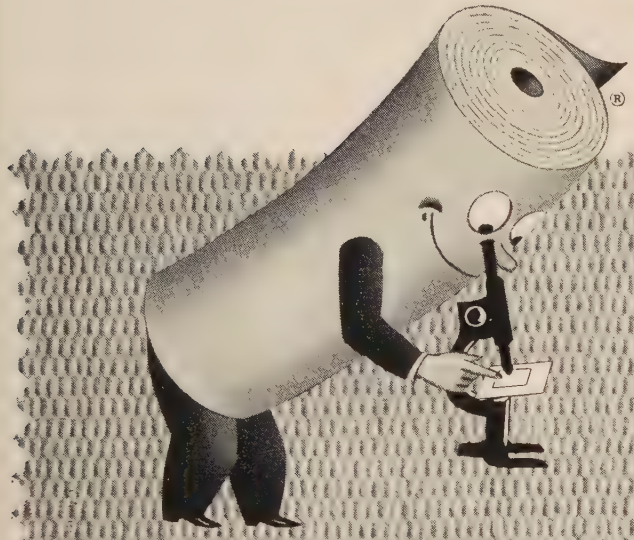

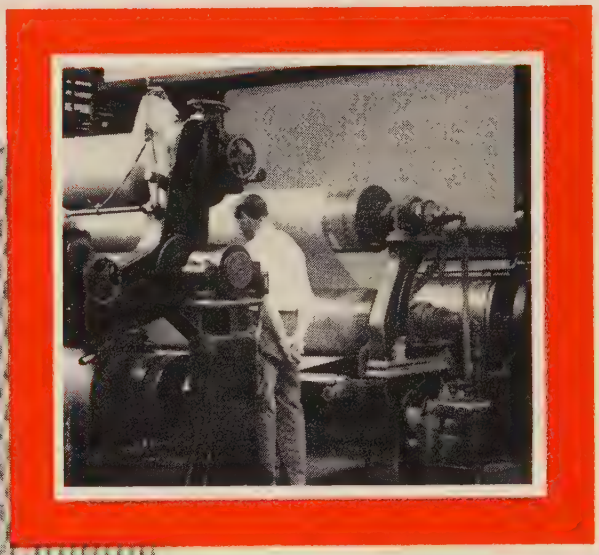

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phate processes and products and a list of common stains for distinguishing one from the other. The progress of the kraft industry in the United States was discussed (*45*, *46*) with the development of the French kraft industry in mind. The outstanding improvements in the kraft industry were reviewed by Schnyder (*47*) under the headings: raw materials; wood and chip preparation; digesters; brown stock washing, screening, and storage; bleach plant; chemical departments; drying and finishing; and utilities. Nolan (*48*) presented a rather detailed description of the variables involved in the sulphate cooking process, including chip size, liquor, time, temperature, size of digester, liquor circulation, etc. Nolan (*49*) also reviewed the pulping research carried out at the University of Florida since 1946. Digester room equipment was described by Whiteside (*50*). Standard digesters, chemipulpers, circulation systems, instrumentation, and corrosion were discussed. The discussion of instrumentation throughout a kraft mill by members of a panel representing mill and instrument companies was described by Schnyder (*51*), who acted as moderator. Kennedy (*52*) presented some ideas on digester instrumentation.

Eucalypts as raw materials in the Australian pulp industry, an example of a typical kraft cook of a eucalypt, and the organization of the Australian industry was presented by Lee (*53*).

The text of the FAO report (*54*) dealing with straw pulping processes has been reprinted (*55*). Williams (*56*) discussed straw and bagasse as raw materials, and recent developments and installations of Celdecor equipment.

## THE COOKING PROCESS

A revision of TAPPI Standard O 400 p-44 (which lists the standard terms used in the sulphate pulping process) has been proposed (*57*).

Tarkkonen (*58*) obtained a patent for using the spent liquor from a completed cook for wetting the chips which are being fed to a fresh digester. It is claimed to help pack the chips.

Simkin (*59*) gave a number of curves and tables of data for commercial kraft cooks of comparatively short duration. The pulp and paper characteristics are given.

Felton (*60*) studied the kinetics of laboratory alkaline cooks and mathematically expressed the variables of temperature and pressure as a "boiling unit," the use of which is claimed to permit easier control in commercial practice.

Nepenin and Orlova (*61*) studied temperature and liquor concentration in kraft cooking in an attempt to decrease the total time. Sender-Lapinska (*62*) examined the maximum temperature, time to maximum temperature, time at maximum temperature, ratio of active alkali to wood, and sulphidity on the production of easily bleachable sulphate pulps from pine.

Fukuda and Mizukami (*63*) compared soda and sulphate cooking, and found that the rate of reaction and consumption of effective alkali was much slower for the former, although at the same yield the amount of lignin, pentosan, and cellulose removed is about the same for both cooks. The degree of polymerization (D.P.) and the mechanical properties of the soda pulp reached a maximum at about 80% yield and then fell gradually with decreasing yield, thus differing from the sulphate pulps where the maximum D.P. and strength occured at yields of 45 to 50%. Soda pulps at this yield were much inferior.

Nolan (*64*) continued his work on continuous alkaline pulping. Using slash pine chips softened by presteaming for 10 min. at 125 p.s.i. and then hammermilled to expand the surface, he reported obtaining pulps in a few minutes which were 95% as strong as the best commercial kraft pulps. A very high (40:1) liquor-to-wood ratio was used to maintain the concentration. Data for a range of concentration from 20 to 80 grams per liter of $Na_2O$ and pressures from 115 to 170 p.s.i. were presented.

A patent obtained by Sloman (*65*) involved a first-stage cook with black liquor to neutralize wood acids, followed by a second-stage cook with fresh white liquor. A saving of 2% in new caustic is claimed. It is applicable to both soda and sulphate processes but particularly the former.

The Va-Purge process was the subject of a United States patent awarded to Ross, Hart, Strapp, and Maass (*66*). A symposium on the process was held (*67*) and included a number of papers of interest to those in the alkaline field. They are: the problem of penetration, by Maass; the Va-Purge impregnation pretreatment: one solution of the penetration problem, by Ross; adaptation of Va-Purge to chemical pulping with conventional equipment, by Hart and Strapp; use of the Va-Purge process in straw pulping for corrugating board manufacture, by Palm and Ostrowski; increase of yield and quality over conventional kraft cooking, by Houghton; the alkaline pulping of mixed hardwoods by Hartford; mill trials of Va-Purge in conventional kraft cooking equipment, by Wilson; and several others. In addition, a number of reviews about the Va-Purge process and the symposium have appeared (*68*).

Jensen (*69*) reviewed the results of a laboratory investigation on the cooking, bleaching, and beating of birch. Jayme, *et al.* (*70*) studied five different species of willow for their suitability for papermaking. The willows, which were 5 to 15 years old, were anatomically similar to poplar, but more difficult to cook under standard kraft conditions, so a temperature of 165°C. had to be used. The yields ranged from 47.7 to 60.15%, being, in general, a bit lower than poplar. The yield was higher in older woods. The strength of the pulp was very high and results indicate that willows are a suitable raw material for paper pulps.

Szlakowski (*71*) made experimental cooks on five species of poplar (*Populus tremula*, *P. alba*, *P. canexens*, *P. berolinensis* and *P. deltoides*). In general, they proved an excellent raw material because of high yields, high strength, low alkali consumption, low maximum cooking temperature (165°C.) and easy bleachability. The last two species mentioned were preferred because of more rapid growth and superior over-all pulp characteristics.

Simmonds, *et al.* (*72*) obtained good dissolving-grade pulp from poplar using a water prehydrolysis followed by a sulphate cook. Purification included chlorination, hot or cold alkali extraction, and treatment with sodium hypochlorite and chlorine dioxide. The ex-

# Experience

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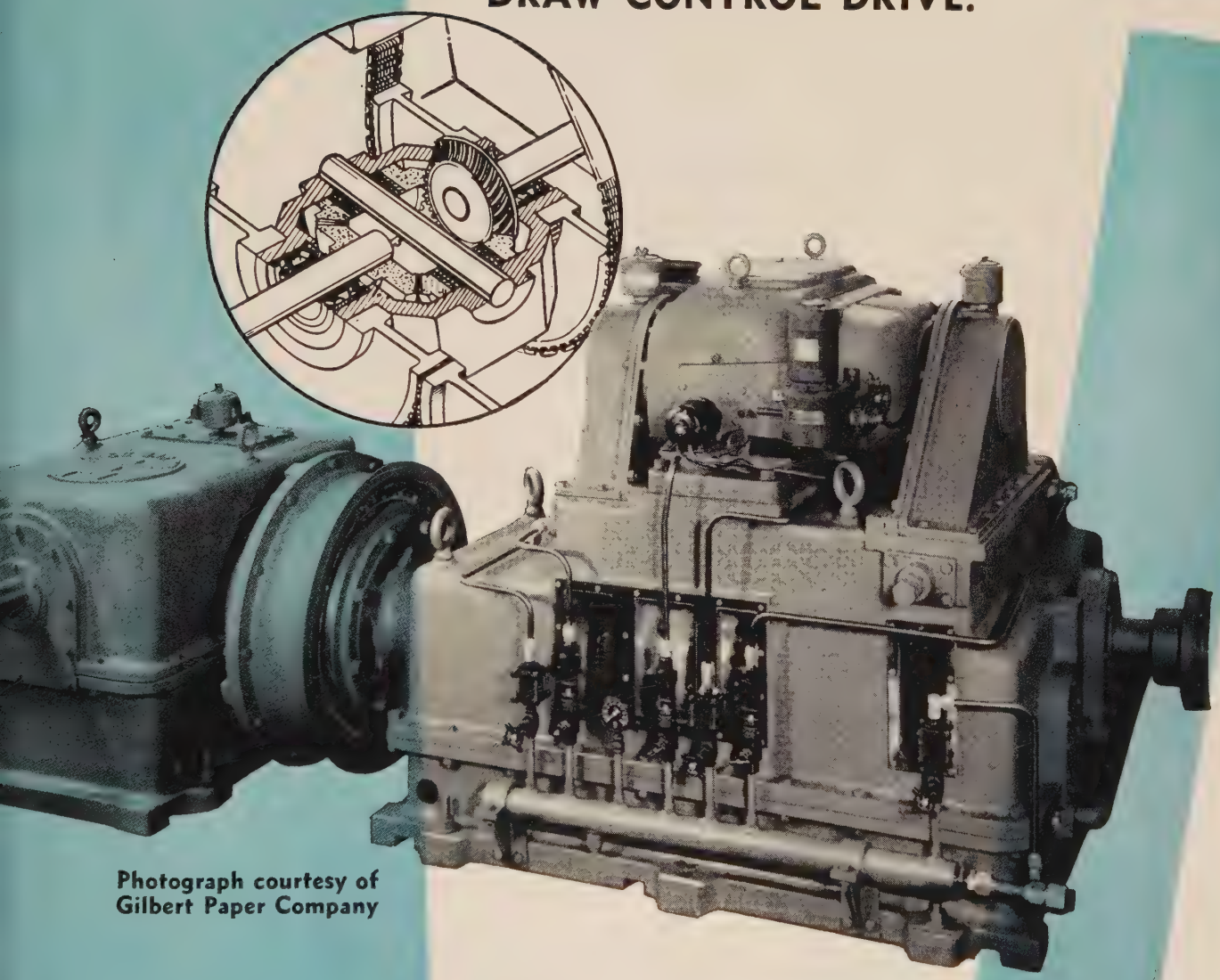

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perimental cellulose acetate yarns obtained were as strong as yarn from a commercial softwood sulphite.

Turner (*73*) investigated the soda pulping of *Eucalyptus gigantea*. He used 170°C. and 0.125, 0.5, 1.0, 2.0, 4.0, and 6.0 hr. for the cook, varying the soda charge to give the same permanganate number in each case. At each permanganate number level the highest D.P. and lowest bleach demand and retention time were found when the time was between $^1/_2$ and 1 hr. The yield was highest at 2 to 3 hr. As cooking time increased from $^1/_2$ to 6 hr., the D.P. steadily decreased at any one permanganate number, whereas bleach demand and retention time increased. The use of the very high soda charge for times less than $^1/_2$ hr. led to marked lowering of the D.P.

Surewicz (*74*) evaluated Murray pine and showed by laboratory cooks that it was very suitable for kraft pulp manufacture, being superior to *Pinus silvestris*.

Solomko (*75*) found that Siberian cedar was suitable for kraft pulping, although it has a high terpene content.

Sender-Lapinska (*76*) described successful commercial runs of newsprint made using (1) 20 to 25% unbleached pine kraft and 75 to 80% spruce groundwood, and (2) 12.5% unbleached pine kraft, 12.5% bleached pine kraft, and 75% spruce groundwood. Such furnishes were recommended for Poland to conserve spruce.

Brown and McGovern (*77*) studied the cold soda cooking of aspen, birch, red alder, sweetgum, cottonwood, red oak, white oak, and mixtures of red alder and the two oaks. One and one-half to two hours at room temperature with 20 to 100 grams per liter of NaOH gave yields of 88 to 90%, the chief loss being of hemicellulose. Pulps could be bleached to a brightness of 65 to 70%.

MacLaurin, Peckham, and Weber (*78*) compared the pulps obtained by the kraft and the neutral sulphite semichemical processes from birch and from maple. The unbleached yields were about 50 to 52% for the krafts, 65 and 70% for the maple semichemical, and 70 and 75% for the birch semichemical. All the pulps were potentially useful. The unbleached kraft pulps were stronger, easier bleaching, and harder to beat than the semichemical pulps. Bleaching lowered the kraft and raised the semichemical strengths, the bleached semichemical being superior to bleached kraft and equal to unbleached kraft in strength. It appeared that for the same pulping conditions birch gave a higher yield of stronger pulp than did maple.

MacLaurin and Peckham (*79*) produced high-yield pulps suitable for corrugating medium by semikraft and neutral sulphite semichemical processes on southern red oak. The yields of pulp by the semikraft process were not greatly affected by changes in maximum cooking temperature, but were greatly affected by increasing the active alkali from 6 to 12%. In the neutral sulphite cooks on the other hand, an increase in sodium sulphite from 6 to 18% decreased the yield only slightly. The neutral sulphite pulps were stronger and lighter colored than the semikraft pulps at the same yield, but they both showed somewhat lower flat crush resistance than a typical aspen neutral sulphite semichemical pulp used for corrugating medium.

Runkel, *et al.* (*80*) compared the pulping of beech with 2.4% NaOH (liquor:wood ratio, 10:1) at 20°C. for 48 hr., 70°C. for 14 hr., 100°C. for 6 hr., and 150°C. for 3 hr. All pulps had about the same lignin content but pentosan content decreased with increasing temperature. All pulps had about the same bursting strength but the 6-hr. cook at 100°C. had the highest tearing strength and the two lowest temperature cooks, the lowest tearing strength and folding endurance.

Saarnio and Gustafsson (*81*) examined the dissolving and destruction of carbohydrates from pine and birch during the sulphate cook. Results indicated that the hexosans which dissolve are rapidly destroyed whereas the pentosans are much more resistant, xylan in particular. However, after 6 hr., most of the dissolved pentosans are destroyed.

Prey, *et al.* (*82*) tried to determine components responsible for the formation of acids during the alkaline pulping of beechwood. The hemicelluloses were shown to be the principal precursors of acids, volatile and nonvolatile in steam, although lignin and alpha-cellulose also make their contribution. The same workers (*83*) continued their study of the consumption of alkali during a cook and concluded that of the alkali consumed by the hemicelluloses during a one-stage soda cook of beech, only one fourth is used for dissolving them and three fourths for neutralizing acids. The degradation of hemicelluloses can only be prevented by low temperatures (not over 100°C.) and pH below 12. However, the dissolution of lignin requires a pH of at least 12 and temperatures above 140°C. The degradation of hemicelluloses can therefore be prevented only by removing them through a mild prehydrolysis before the cooking. The investigations appeared to show that by increasing the removal of hemicellulose below 100°C., the yield of alkali lignin could be reduced to one half the amount obtained under standard cooking conditions, and the authors conclude that at least a portion of the technical alkali lignin obtained from normal cooks is formed from the hemicelluloses.

Berthier (*84*) studied the cooking of Maritime pine with sodium polysulphide, comparing it with sodium sulphide. At the same chlorine and permanganate numbers, the polysulphide gave a stronger pulp in higher yield, and the superiority was maintained through bleaching. The cellulose had a higher average D.P. The result is correlated with the fact that polysulphide decomposes to sulphide at cooking temperatures and the sulphide concentration is thereby maintained. Disadvantages are a higher consumption of chemical and certain corrosion problems.

Hägglund (*85*) prepared various thiolignins with sulphur contents from 3.7 to 12%. In those with low sulphur percentages the sulphur may be present as a thio ether, whereas in the high sulphur-containing thiolignins the sulphur is largely present as disulphide. The removal of carbohydrate material during an alkaline cook is due to the degradation of enolizable end groups, since methyl glucoside is stable under these conditions.

Zentner (*86*) studied the reaction products of lignin model compounds and sodium hydrosulphide under conditions similar to those pertaining in a kraft cook. In addition to the guaiacyl nucleus, the compounds contained carbonyl groups, conjugated double bonds, ether linkage, and aliphatic hydroxyl. Only the last reacted to form a sulphur derivative. Small yields of de-

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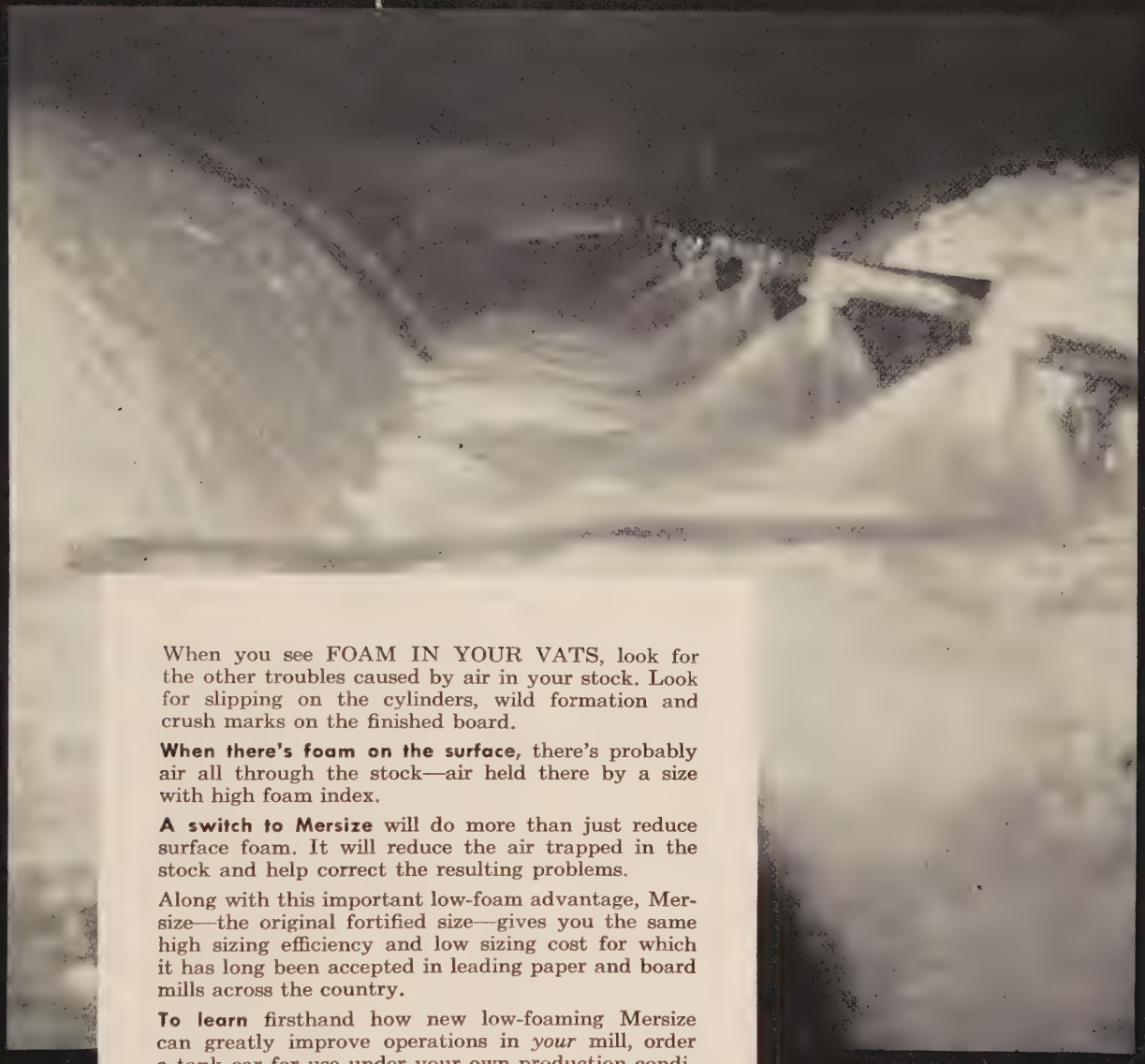

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methylated products were isolated from the cooks of all compounds containing a carbonyl group. The conjugated double bonds of three different alpha beta unsaturated ketones were hydrolyzed by the cook. Cleavage of the ether linkage of one model compound was accompanied by a reducing reaction. The results pointed to a possibility that hydroxyl groups in lignin react during a kraft cook to form thiolignins.

Cottrall (*87*) reviewed the influence of chemical composition and morphology of fibers of mechanical and kraft pulps on their papermaking properties. Ribbonlike, flexible fibers are more desirable for printing and wrapping grades where high burst and tensile are required, whereas the solid fibers from southern pines, Douglas-fir, and western hemlock are satisfactory for papers in which tear resistance, lightness, porosity, and bulk are desirable features. In many instances blends are most satisfactory.

Higgins and Lewis (*88*) evaluated bleached kraft pulps for boxboard on the basis of factors important in both manufacture and conversion—i.e., time required to reach a freeness of 300 to 400 ml. Canadian, scoreability, and printability. Pulps from different parts of the United States were studied. Those from northeastern species were smoother than those from southern or northwestern krafts.

Japanese workers (*89*) studied the production of a rayon pulp from *P. densiflora* by the kraft process without prehydrolysis. The emphasis was on reactivity of the pulp and filterability of the viscose rather than on the elimination of pentosan. The pulp from a conventional kraft cook (D.P. 1600 to 2100) was treated with chlorine water at pH 1 to 2 at room temperature for an hour, then heated at 100°C. for 1 to 4 hr., refined with alkali, and bleached with hypochlorite or chlorine dioxide. Pulp in good yield (91 to 92% alpha-cellulose and 700 to 800 D.P.) was obtained, and it compared well in filterability with prehydrolyzed pulps. The spinnability and quality of filament were not affected.

There is mention of two Japanese patents (*90, 91*) on the production of rayon pulp by the kraft process using presteaming at 150 to 170°C. prior to the cook.

***Agricultural Residues, Grasses, Tropical Hardwoods, etc.***

At the Netherlands Experimental Station for the utilization of straw, Asselman, *et al.* (*92*) have studied the cooking of rye straw with caustic soda. The time was held constant at 4 hr. while the NaOH concentration, temperature, and straw:liquor ratio were varied. The latter was found to be relatively unimportant. The best over-all result was probably at 130°C. with 13% NaOH, but for optimum tear resistance the temperature should be not lower than 160°C. and NaOH concentration not less than 15%. For optimum fold a temperature of 145°C. was necessary.

Hebbs (*93*) discussed straw pulping in the United Kingdom and gave reasons why esparto grass is preferred. With the latter, more material can be charged into a digester, less caustic is required, drainage is better (leading to better liquor recovery), yields are higher, and spent liquors have a higher calorific value. A defect in straw which is not evident from laboratory investigations is the tendency for it to give small transparent specks in heavily calendered sheets.

At the Trenton, Ont., mill of Hinde & Dauch, they have changed over to the Va-Purge process (*67, 94*). At the same time a change from lime to caustic soda has occurred and it is reported that the yield of straw pulp has increased from 60 to 70% and the time reduced 30%. The harder pulp permitted a speed-up of the paper machine and a reduction of the basis weight.

Jayme and Scheuring (*95*) published the initial paper in a series on the production of acetate pulps from cereal straw. Using prehydrolysis, a kraft cook, and multistage bleaching, they obtained yields of around 30% from rye and wheat straw. The pulp was easily acetylated but produced hazy solutions. When the bleaching was followed by a cold 10% and a hot 10% caustic soda extraction, clear acetate solutions were obtained. The most satisfactory rye straw pulp was obtained in 27.6% yield, had a brightness of 89.1 (against barytes), 98.5% alpha-cellulose, 0.21% ash.

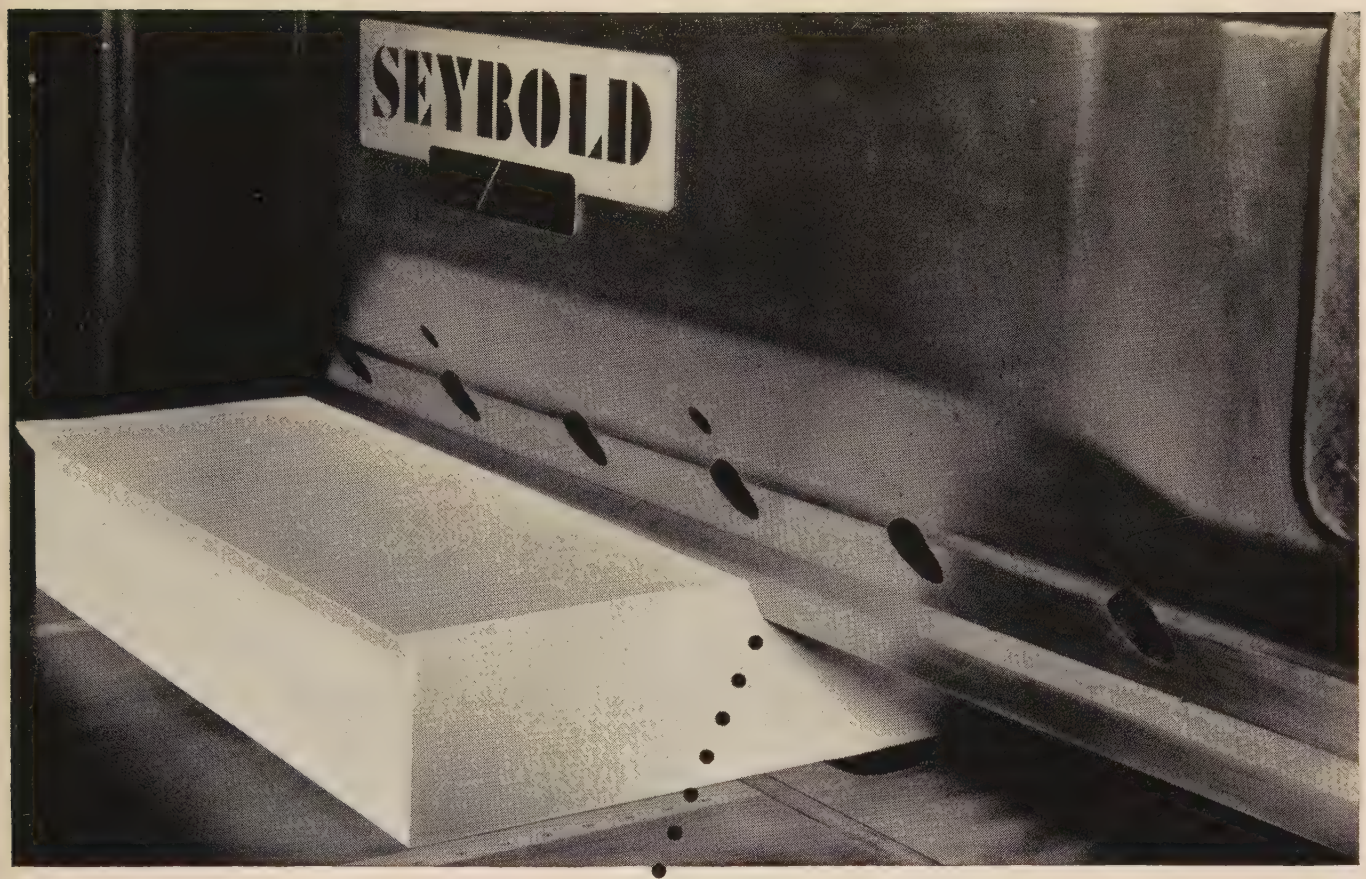
Jayme, Branscheid, and Harders-Steinhäuser (*96*) published the results obtained during the war on the production of dissolving pulps from the reed *Phragmites communis*. Trin. The fibers are short and thick-walled, making them unattractive for papermaking but a satisfactory dissolving pulp was obtained by a sulphate cook following prehydrolysis. The yield was 28.38%, alpha-cellulose 91.5%, pentosan 1.79%, ash 0.13%.

Perila (*97*) compared the pulping of bagasse by a number of established methods, the object being to find which produced an easy-bleaching pulp with the minimum consumption of chemicals. Of the sulphate, Pomilio, sodium sulphite, sodium bisulphite, combined sulphite-bisulphite, and prehydrolysis-sulphate, the first three were best. A patent for an eight-stage process for producing dissolving pulps or fine quality papers from bagasse was issued to Atkinson and Rogers (*98*). The stages are (1) mechanical disintegration and washing, (2) 2% nitric acid at 80 to 110°C. for 1 hr., (3) alkaline extraction with 1 to 3% caustic soda at 95°C. for 1 hr., (4) mechanical agitation, (5) repeat of (3), (6) chlorination, (7) alkaline steep, and (8) rebleach with chlorine, etc. For producing newsprint from bagasse, Bhat (*99*) states that the rod mill is better than a Kollergang or defibrator for removing pith. A pulp made by cooking bagasse with 10 to 20% NaOH at 142 to 162°C. for 3 to 6 hr. was mixed with 30% bamboo pulp and the paper run successfully on the machine.

A patent was issued to Van Dyk (*100*) for a method of obtaining fibers from undecorticated ramie, jute, hemp, seed flax, etc. It consists of cooking for an hour at 15 to 45 p.s.i. in a solution of 2% alkali metal silicate, mixed ethanolamines and oleic acid, buffered to pH 12.4 to 13.0.

Wells (*101*) has patented a process for obtaining fibers from seed flax, and with reference to a previous patent (*102*), he states (*103*) that a cold caustic treatment of the fluff obtained from rod-milling, followed by Bauering, passage through the Vortrap, and bleaching, gives a pulp suitable for fine paper.

Pustelnik and Winczakiewicz (*104*) found that a useful pulp could be produced from the flax waste obtained from carding machines by cooking for an hour at 130°C. with 3% NaOH, Kollerganging at 22 to 24% consistency for 2.5 to 3.5 hr., washing, and bleaching. The

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pulp was used successfully in the manufacture of cigarette paper.

Bhat and co-workers have continued their work in India on the pulping of indigenous plants. Ulla grass (*Themeda arundinacea*, Ridley)(*105*) produced a good long-fibered bleached pulp in satisfactory yield by the soda or sulphate processes. Panni grass (*Vetiveria zizanioides*)(*106*) gave a soda pulp suitable for writing papers but the bleach consumption was high. Kusal or spear grass (*Andropogon contortus*, Linn)(*107*) gave satisfactory pulps by sulphate or soda processes, but the former was preferable due to higher yield, better strength, and lower bleach consumption. Papita (*Sterculia campanulata*, Wall)(*108*) gave easy bleaching sulphate pulps in good yield under milder conditions than is general for other woods. The average fiber length of 1.49 mm., which is similar to esparto, makes it suitable for writing and printing papers. The small, fast-growing, short-lived tree, *Trema orientalis*, Blume gave bleached sulphate pulps in 46 to 50% yield (*109*). The fibers are 1-mm. long on the average.

Pustelnik and Bittmar (*110*) evaluated a number of annual plants using the kraft process. The pulps were satisfactory from *Ricinus communis*, *Papaver somniferum*, *Camelina sativa*, *Althaea officinalis*, *Althaea rosea*, *Carthamus tinctorius*, and Albanian rush.

Chittenden, Morton, and Pickering (*111*) pulped Batai wood (*Albizzia falcata* L.) from Malaya by the sulphate process and obtained pulps in 51% yield comparable to conifer kraft except for lower tear. The length of the fibers is 1.18 mm.

## SULPHATE LIQUOR CYCLE

Brown (*112*) reviewed the kraft liquor cycle from an operator's viewpoint, discussing the equipment, its layout, personnel requirements, the operation and its control, and maintenance. Corrosion in the recovery plant was reviewed by Teeple (*113*) who dealt with the recovery furnace, boiler, dissolving tank, liquor spouts, induced draft fans, salt-cake mixing tank, disk evaporators, and duct work, making suggestions for suitable materials of construction. The losses of alkali which occur in a kraft mill, both accidental and normal, were discussed by Nepenin and Orlova (*114*). They calculated that the loss (as $Na_2O$) during cooking, washing, evaporating, burning, and causticizing was 0.28, 6.67, 4.91, 18.77, and 10.03 kg. per ton of oven-dry pulp, respectively.

A review of recent recovery furnace installations by Hochmuth (*115*) indicates that there has been no basic change since the vertical tower type appeared in 1937–40. In the past 10 years there have been many improvements on this basic design, resulting in a reduction of labor and maintenance. There is a trend toward large capacity single-stage units, and operation at higher steam temperatures and pressures, several now being operated at 825°F. and 850 p.s.i. One of the most recent developments is an improved superheater which is less susceptible to slag accumulations. Advances in the design of Babcock and Wilcox furnaces were reviewed by Darmstadt (*116*). They include steam shatter jets in the smelt spouts to reduce noise, automatic air flow control, and all-retractable soot-blowing system, and the Pease Anthony venturi fume scrubber. Studies of recirculating some of the flue gas to the char bed indicate some advantages in temperature control.

Upson and Johnson (*117*) described the instrumentation of the recovery system at the Savannah plant of the Union Bag & Paper Corp. Spent liquor preparation, combustion, recovered chemical removal, steam generating equipment, soot-blowing equipment, and fume recovery instrumentation are treated separately.

The systems used in cleaning recovery boilers have been described by Walleze (*118*). Retractable-type blowers appear to be gaining favor. Both steam and air are effective, although the latter is easier to maintain. Blowers should be able to use both.

A review of the operation of the Tomlinson secondary recovery system at the Canada Paper Co. was given by Allan (*119*). It recovers chemical and heat from the effluent gases from a new 175-ton B & W Tomlinson furnace, producing 100 lb. of sodium salts and $4 \times 10^6$ B.t.u. per air-dry ton of pulp.

A Canadian patent has been issued to Tomlinson & Tomlinson (*120*) for a cyclic black liquor recovery system which produces substantial amounts of hot water and is claimed to have high thermal and chemical efficiency. It is the same as a previous United States patent.

A Canadian patent, similar to a previous United States patent, was awarded to Ely and Rogers (*121*) for a spray tower to recover heat and chemicals from waste gases.

A vibrating screen for feeding salt cake into the mixing tank has been patented in Canada by Hochmuth (*122*). This had previously been patented in the United States.

Other Canadian patents previously obtained as United States patents are that of Gagliardi, *et al.*(*123*) for adding a low ash, sulphur-containing coke to the black liquor prior to burning, and that of Nilsson (*124*) which relates to black liquor evaporating and burning.

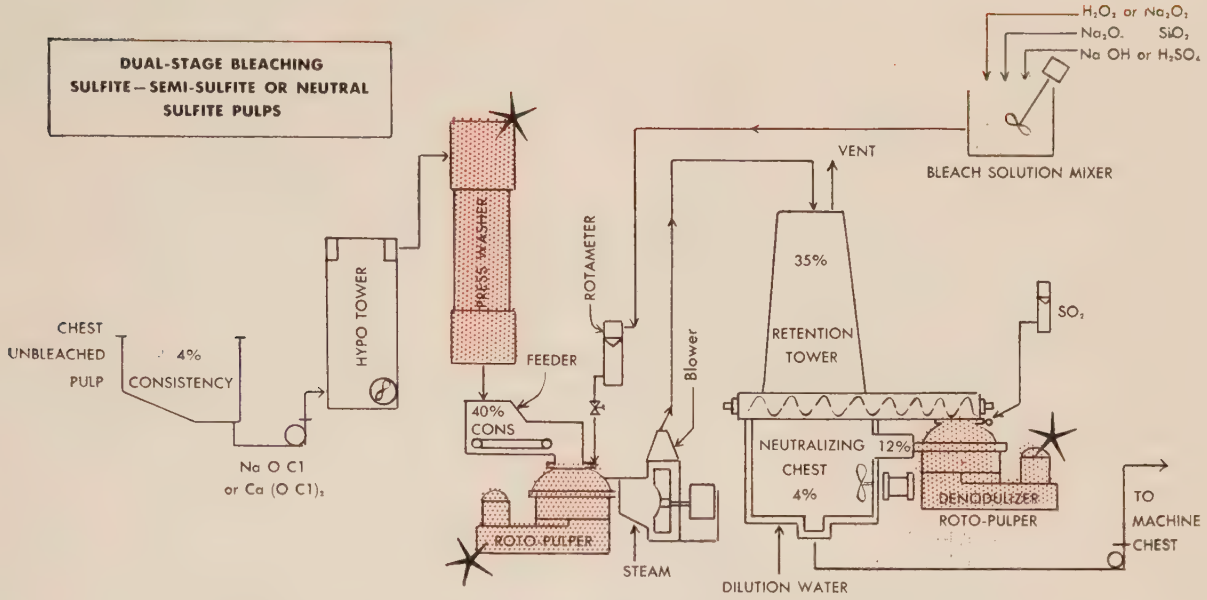
An apparatus for discharging a horizontal flat spray of liquor just above the hearth of the combustion furnace has been patented by Singleton (*125*).

Aries and Pollak (*126*) patented a system for adding molten ash to a concentrated solution of sodium sulphide. The carbonate and sulphate have a limited solubility in this solution, settle out, and are easily separated. Water is added continuously as the ash is added and concentrated sulphide solution continuously withdrawn.

Vassie (*127*) discussed advantages to be gained from adding liquid sulphur to the recovery furnace instead of lump sulphur. These were reported as a higher efficiency of burning and less labor.

The relationship between fuel value, ash content, and water content of sulphate and sulphite liquors has been established by Sadler, *et al.* (*128*). In laboratory experiments on the cooking of spruce by both processes, the spent liquors, including all wash waters, were separated, concentrated, and the calorific value determined. Under normal conditions the sulphate liquors have more inorganics and up to 30% less fuel value. Under drastic cooking conditions the sulphate liquors have more organics and a higher fuel value than sulphite liquors.

# J-C dual-stage* bleaching

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### *Evaporation*

A review of the 1952 literature on the theory and practice of evaporation has been made by Badger and Lindsay (*129*). One section deals with the evaporation of black liquor and its problems. Foulon (*130*) discussed modern evaporators and emphasized that the use of vacuum does not effect savings in steam but is essential where only low pressure steam is available. A Japanese patent for the concentration of black liquor *in vacuo* was issued to Kameda and Tsuzuki (*131*). The liquor is treated with a mixture of an equal weight of higher alcohol and paraffin to prevent foaming during evaporation.

The construction and operation of two commercial direct contact evaporators was described by Reid (*132*). They are the B & W cyclone scrubber and the D. J. Murray cascade evaporator.

A cyclone evaporator for concentrating kraft black liquor by direct contact with flue gases from the burning of the concentrated liquor is the subject of a Canadian patent awarded to Tomlinson and Swartz (*133*). It is the same as a previous United States patent.

A system for concentrating black liquor by the submerged combustion of gas or oil was described by Weisman (*134*) as having low capital cost and maintenance but it has a high fuel consumption compared with multiple effect evaporators. It is probably uneconomical for concentrating up to 45% solids, but might be suitable for further concentration up to 65% solids.

Hall (*135*) issued a progress report on the work being carried out on the plugging of long-tube, vertical, multiple effect evaporators. It is concluded that the plug is probably composed of black liquor solids rather than sulphate soap, and may be initiated by scale formation induced by the use of superheated steam.

Hedström (*136*) compared the corrosion behavior of pure carbon steel with 5% nickel steel in the tubes of black liquor evaporators, and reported that the latter is more effective the more aggressive the corrosive conditions. Lindh (*137*) discussed the use of stainless steels in the Swedish pulp industry and indicated that 13% chromium steel tubes in the first two effects of black liquor evaporators where corrosion is heaviest, have proved very successful in Swedish and Finnish mills. This alloy is cheaper than the 18/8 steel commonly used in the United States and Canadian mills.

The specific heat of black liquor has been measured over the concentration range 4.9 to 52.6% solids and temperature of 100 to 200°F. by Harvin and Brown (*138*). They developed an empirical equation relating specific heat, percentage solids and temperature, and used it to extrapolate to higher concentrations and temperatures.

### *Analysis of Black Liquor*

Phillips and Rubright (*139*) described the use of the Karl Fischer reagent for determining water in pulp mill liquors and hence by difference the percentage of nonaqueous constituents. In contrast to oven-drying methods, it only takes a few minutes.

Borlew and Lancaster (*140*) determined black liquor solids by infrared drying, and compared the results with the oven-drying and xylene distillation methods. It is considered to be at least as accurate as the others, is rapid, and uses a smaller sample. It measures the sum of water and other volatiles so that by using the Karl Fischer reagent for water it would be possible to calculate the nonaqueous volatiles.

A letter to the Editor of the Technical Association of the Pulp and Paper Industry (TAPPI) by W. E. Bryden (*141*) requested information concerning the analysis of black liquor for carbon, hydrogen, oxygen, sulphur, nitrogen, and ash. A discussion and suggestions followed in the replies of Kennedy, Pineo, Ervin, Limerick, Yundt, and Browning.

A new method for the determination of sulphides in sulphate black liquor was described by Bethge (*142*). It is based on the formation of molybdenum blue when a solution of molybdate and sulphite in phosphoric acid is added. The absorbency of the centrifuged solution is measured at 700 mmu and is proportional to the sulphide content.

Dzhelyuk (*143*) studied three methods for the volumetric determination of sulphate in kraft liquors by precipitation with standard barium chloride followed by back titration of the excess barium salt. He concluded that the accuracy increased in the order sodium carbonate, chromate, phosphate.

### *Black Liquor Oxidation*

Collins (*144*) made a comprehensive review of the literature under the headings: advantages of oxidation, Bergstrom-Trobeck system, corrosion studies, Tomlinson studies, INKA evaporating system, Bialkowsky and DeHaas studies, Wright's research, work of Collins, other studies, Mannbro analysis. Advantages of oxidation listed by Laszkiewicz (*145*) are less loss of sulphur, lower lime consumption in causticizing, reduction of odor, reduced corrosion, better heat recovery. One disadvantage noted was the higher viscosity of the liquor below 60°.

The experience of a Norwegian mill in using the Trobeck oxidation system was described by Sylwan (*146*). The capacity of the system increased, there was a saving in fuel, less loss of sulphur, and lower lime requirements. For optimum results it is necessary to remove as much as possible of the soap before the liquor goes to the tower.

A Canadian patent, previously issued as a United States patent, was awarded to Kress (*147*) for an oxidation system. From the top of the tower the liquor cascades down over inclined shelves while air passes up.

Wright (*148*) described the effect of the type of packing on the rate of black liquor oxidation. Actual tower packing materials were used rather than scaled down approximations and the liquor was recirculated to approximate full-scale conditions. It was shown that vertical sheets having a suitable surface pattern had advantages of improved oxidation with no foaming.

### *Odor Control*

Wright, *et al.* (*149*) described a very compact experimental unit using the above-mentioned type of packing which effected substantially complete oxidation of the sodium sulphide in the liquor with no foaming. Spent air from the unit had no detectable amount of hydrogen sulphide or methyl mercaptan. Digester gases, after passing through a condenser, may be added to the air entering this unit, with significant retention of sulphur and no loss of oxidation efficiency.

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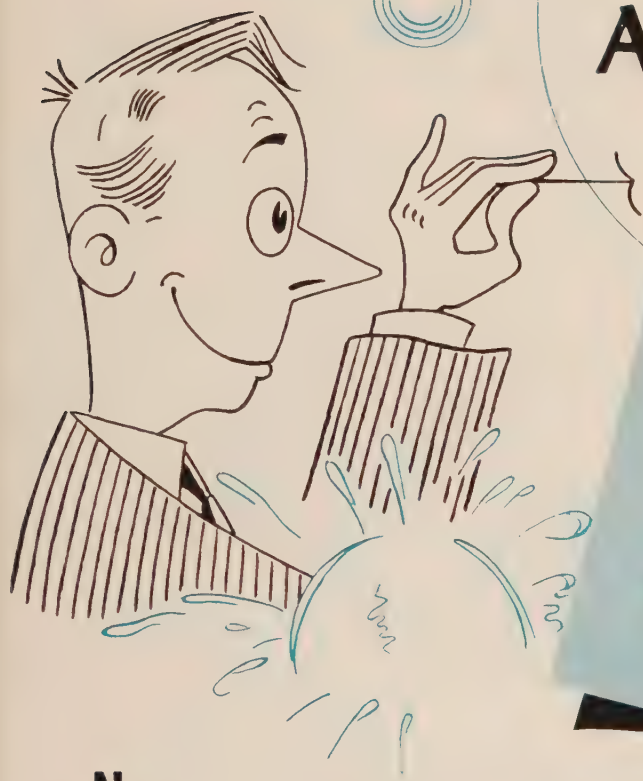

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Wright, *et al.* (*150*) analyzed the various effluents from the recovery system of a mill which was fed exclusively with oxidized black liquor for 5 hr. Results were encouraging in that the amounts of hydrogen sulphide and methyl mercaptan set free were reduced. However, it is felt unlikely that odor release will be reduced to zero, and since any perceptible odor is disagreeable, it is probable that the most that can be hoped for is a reduction of the area over which the odor can be detected.

Cate (*151*) discussed the design of chimneys for the dispersion of odors. By increasing the height the odor at ground level can be reduced and proper chimney design may be useful. However, it is felt that more positive means are needed.

The experiences of the Gulf States Paper Corp. were presented by May (*152*) in chronological order. Condensing the blow gases and the use of chlorine for deodorizing were tried, but without success. Trials of odor masking agents started in 1951 and showed slight benefits but more work is needed for positive conclusions. A great need was felt for a practical and accurate method of measuring odor.

Woodward (*153*) suggested that chlorine dioxide should be tried since it has been used successfully in other industries.

Meuly and Tremaine (*154*) described the results of laboratory studies and extensive field trials with odor masking agents. Semiquantitative odorimetric measurements were made on the basis of a number of special units: scent unit (U), scent value (SV), gram scent value (GSV), mask unit (MU), mask value (MV), and masking ratio (MR). They permit an evaluation to be made of the intensity of kraft mill odors and the relative effectiveness of various masking agents. In this and a subsequent paper by Tremaine (*155*) the application of odor masking agents by (1) spraying, vaporizing, or atomizing into air-gas streams, (2) adding directly to the process, (3) adding to scrubbing liquors, (4) spreading or floating on contaminated surfaces without dilution, is described. In the pulp industry the second system appears to be the best, the masking agent being added directly to the digester after filling with chips and liquor. The influence of weather conditions was also discussed.

McCormick (*156*) reported that the mill trials of odor masking agents at the Pasadena, Tex., and Canton, N. C., mills of the Champion Paper & Fibre Co. showed that up to 85% of the malodors could be eliminated. The agent was added directly to the digesters.

A method for the quantitative determination of methyl mercaptan, dimethyl disulphide, and dimethyl sulphide in a gas mixture was described by Segal and Starkey (*157*). The gas is passed through a solution of mercuric cyanide and then benzene. The former absorbs mercaptan whereas sulphides are removed by the latter. The mercaptan can be determined by volumetric methods or by weighing the insoluble mercuric dithiomethoxide precipitate. In the benzene solution the disulphide is reduced to mercaptan which is then determined titrimetrically after reaction with silver nitrate. The sulphide is determined by titration of the hydrobromic acid liberated upon reaction with bromine.

A method of increasing the yield of methyl sulphide from black liquor has been patented by Hägglund and Enkvist (*158*). An inorganic sulphide or hydrosulphide is added to the liquor and heated to 150 to 500°C. The mercaptan formed is then converted to methyl sulphide by passing over metal sulphide catalysts, or by saturating with alkali salts of methyl sulphate. In an example, the yield is 29 kg. per ton of pulp.

A review of the kraft mill substances responsible for the pollution of air and water was made by Bergström (*159*) who discussed the locations where they are discharged and the methods for eliminating or rendering them harmless. An English translation (*160*) of this Swedish article appeared later.

#### *Turpentine*

According to a Swedish patent awarded to Kulvik and Hannunkari (*161*), the amount of turpentine recovered is increased and recovery is facilitated by blowing off the turpentine-containing gases from one digester and leading it into the bottom of another which is being filled or has just started up.

#### *Causticizing*

Gillespie (*162*) reported the results of a field survey of 29 southeastern mills concerning the operation of continuous recausticizing systems. The over-all operation appears to be satisfactory, but the slaking operation, diaphragm pump maintenance, and scaling of lines are phases which need attention. Suggestions for improvements are made. Two variations of the standard flowsheet are described: two-stage slaking which helps the hot lime handling, and two-stage causticizing which leads to higher causticizing efficiencies.

Kobe and Wilkinson (*163*) studied the effect of sodium sulphide, sulphite, and thiosulphate on the causticizing of carbonate. Sodium sulphite has a greater effect on the equilibrium causticization of carbonate than does sodium sulphite, despite the fact that the former is causticized while the latter is not. However, the amount of sulphite in kraft green liquor is small and being removed by causticization its effect is usually not detrimental. For the concentrations of sulphide normally found in green liquor the causticization of carbonate would be decreased only about 1 to 2%. Sodium thiosulphate does not react with lime and does not affect the causticizing equilibrium.

Venemark (*164*) described the causticizing experiences at a Swedish mill. Potato starch in the amount of 0.1 kg. per ton of pulp is still added to hasten precipitation of calcium carbonate, although there is doubt concerning the value of this treatment. He studied the influence of impurities in the lime and concluded that the method of slaking is more important than the absence of impurities. Equally good causticizing results can be obtained with fresh and reburnt limes.

A patent obtained by Gruen (*165*) concerns the recovery of spent liquors from the cooking of material containing silica. The silica is removed by precipitating with lime before evaporation, the best conditions being higher temperatures (up to and above atmospheric pressures) and over a period of 5 to 10 min. —this permitting maximum precipitation of silica and a minimum of organic matter.

A method for recovering hydrogen sulphide from

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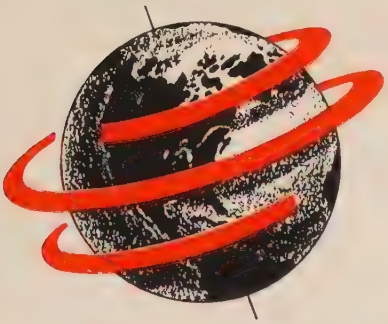

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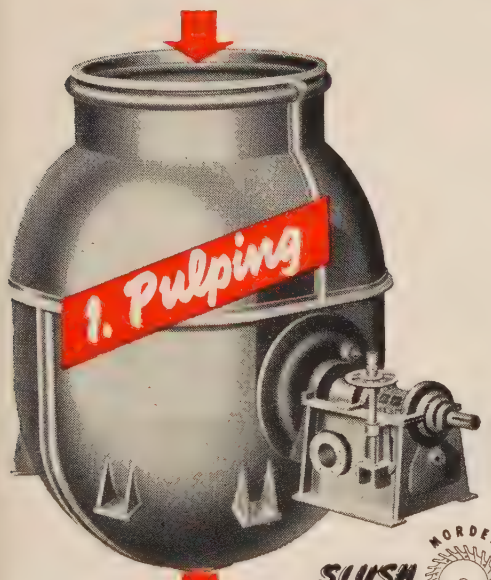


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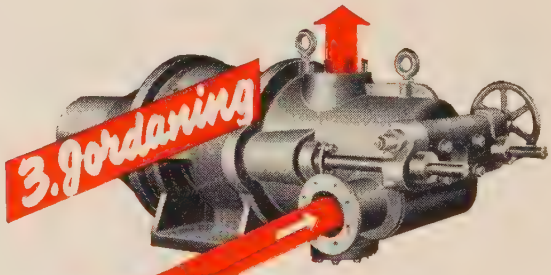


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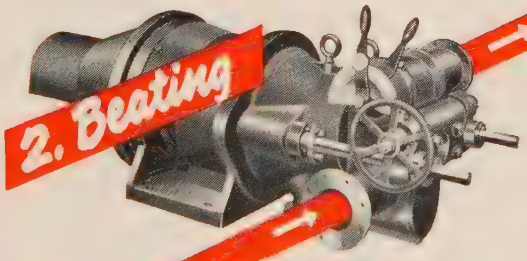


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sulphide-containing soda liquor has been patented in Canada by Mannbro (*166*)(previous United States patent, 1952). A solution of the smelt and a carbon dioxide-containing gas are passed in countercurrent flow through an absorption tower.

Borlew (*167*) discussed the double titration control test for determination of the principal constituents of the green and white liquors, and the variety of interpretations which are possible from the resultant data.

Kahn (*168*) obtained a Canadian patent (previous United States patent) for the clarification of white liquor by centrifuging and coagulating the turbid filtrate with a magnesium compound.

## STREAM POLLUTION

The National Council for Stream Pollution issued its 1952 report (*169*) on the research and development activities being carried out by various institutions. These institutions and programs include Louisiana State University (kraft mill wastes); Virginia Polytechnic Institute (semichemical wastes); Rutgers University (white water, rag, rope, and jute wastes); University of Michigan (stream analysis); Purdue University (strawboard wastes); The Institute of Paper Chemistry and Oregon State College (aquatic biology). Other colleges deal with sulphite and deinking wastes. The project at Louisiana State University was discussed by Moggio (*170*). The most significant achievement to date is the development of an accelerated biological process for treating kraft mill effluents based on the addition of treated wastes to larger volumes of untreated wastes. A greatly accelerated oxidation is obtained. Current studies involve the removal of color with hydrated lime. The same project at Louisiana State University was described by Pesch (*171*), who also reviewed general developments in the field of kraft mill waste reduction, black liquor recovery, reduction of washer and evaporator room losses, and fiber losses.

Moggio and Gehm (*172*) described a pilot plant which was set up in 1951 at Macon, Ga., to test on a larger scale the results of previous laboratory work on the use of activated sludge to treat kraft mill wastes by biological means. Construction was financed by a group of southern kraft mills and operation and maintenance are financed by the National Council for Stream Improvement and the Macon Kraft Co.

Murdock (*173*) discussed the application and limitations of the three types of lagoons in use: aerobic, anaerobic, and chemical treatment. The successful operation of an anaerobic five-lagoon installation at a a strawboard mill is described.

Van Horn (*174*) outlined the stream pollution problems arising from sulphate, semichemical, straw pulping and deinking operations, with emphasis on reduction of B.O.D.

Jung (*175*) described laboratory experiments on strawboard mill wastes which showed that by adding a digested sludge from domestic sewage to mixed mill wastes, an anaerobic flocculent sludge is produced which settles easily. The B.O.D. was reduced by about 40%. The results were confirmed in a semicommercial-scale plant.

A review of recent developments in the treatment of pulp and paper mill effluents from esparto, straw, and rag pulping, deinking and paperboard operations, was made by Horlock, *et al.* (*176*). Recent trends are toward high-rate treatment methods for both suspended solids and B.O.D. removal.

Harwood (*177*) discussed the treatment of effluents with aluminum sulphate and a mixture of this with sodium aluminate.

Wilson (*178*) described the effect of a kraft mill's waste on the bottom fauna of the Clearwater and Snake Rivers in Idaho. Although the dissolved oxygen and B.O.D. were little affected, the presence of basic substances such as sulphides, mercaptans, and resin acid soaps, and of carbohydrates which stimulate sewage fungi, rendered conditions untenable for most bottom fauna normally to be expected in such a river.

Haydu, Amberg, and Dimick (*179*) examined the effect of the volume of test solution on the lethality of methyl mercaptan to king salmon, and concluded that an increase in volume resulted in a decrease of the concentration of mercaptan necessary to give 100% mortality.

A review of the kraft mill substances responsible for the pollution of air and water was made by Bergström (*159*) who discussed the locations where they are discharged and the methods for eliminating or rendering them harmless. An English translation (*160*) of this Swedish article appeared later.

Bruneau (*180*) reported the results of an investigation by the Swedish Department of Fisheries on the pollution of coastal waters in the vicinity of pulp mills—sulphite and sulphate—located in different coastal and estuarial areas of the province Ångermanland. The chief purpose of the study was to examine the distribution and flow of the pollution at various distances from the mill, comparing the conditions existing in 1949 and 1952. There was an improvement in the latter year, probably due to the spent liquor evaporation program instituted by the Mo och Domsjo mills.

## DIGESTER CORROSION

A review of United States, Canadian, Swedish, and Australian work on alkaline digester corrosion was made by Teeple (*181*). Teeple (*182*) also reported to the TAPPI Digester Corrosion Subcommittee the results of a questionnaire sent to 48 kraft and 15 soda mills in the United States. The returns showed that many more mills now than in 1950 are making Audigage or similar thickness measurements and are prepared to provide the committee with their accumulated data. Shoumatoff (*183*) reviewed the activities and projects of this subcommittee. The major current project is that of the Armour Research Foundation, the status of which and plans for the future were discussed by Caywood (*184*).

The uneven corrosion experienced in digesters was studied by Flynn, Richter, and Snyder (*185*). The digesters were lined with type 405 alloy and it was found that some areas were attacked while others were not, but were covered with film and scale. It was decided that splashing of liquor against the hot metal surface during filling was a major cause of the observed corrosion pattern and two cures were tried. One involved filling from the bottom to avoid splashing, while the other consisted of mounting a specially designed liner $1/2$ in. away from the digester shell. The modifications

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proved beneficial, and the Crown Zellerbach Corp. installed two stationary "rimmed steel" digesters with bottom liquor charging to avoid the "hotplate boiling action" form of corrosion (*186*).

Some observations on the causes and prevention of corrosion were made by Hopper (*187*). Carbon steel was satisfactory up to 1934–35, but thereafter the life of a digester was only 1 to 2 years and he outlines the many factors, apart from the steel itself, which might be responsible. Lientz (*188*) placed part of the blame for modern kraft digester corrosion on the changes in steels which have occurred. He reviewed digester corrosion research news. A review of the subject by Guba (*189*) included the comparative costs of stainless steels and he indicated that cost and corrosion resistance are closely related. A new corrosion-resistant alloy, Hastelloy Alloy F, was described by Scheil (*190*) who presented data to show that it might have desirable properties for alkaline service. Baisch (*191*) described the experiences of a mill which applied $^1/_8$-in. thick Inconel strip to two tumbling digesters. Excellent corrosion resistance was found, but excessive cracking occurred in the welds and improvements in fabrication techniques must be sought.

Bukowsky (*192*) stated that corrosion started in the Port Townsend digesters of Crown Zellerbach when the brick-lined recovery furnaces were replaced by chrome-ore linings, and became greater when western red cedar was used in place of Douglas-fir or western hemlock. Stainless steel 347 with columbium was used to make a $^7/_{16}$-in. lining, but serious stress corrosion developed. The upper quarter of the digester was then overlaid with Inconel and this was found to be more resistant, the rate of corrosion being 0.008 i.p.y. in the vapor zone and none elsewhere.

MacLean and Gardner (*193*) have studied the effect of heartwood extractives on digester corrosion and shown that organic complexing or sequestering agents formed in the cook or extracted from the wood may be important. In the case of western red cedar the thujaplicins react with metal only in the absence of alkali and are therefore only important during steaming and filling, but another fraction of the extractives, phenolic in nature and possibly related to the catechol derivative found in the heartwood of Douglas-fir, was also shown to be corrosive.

In a survey of Norwegian sulphate mills described by Roald (*194*), visual inspection of scale and corrosion patterns and Audigage thickness measurements were made. Corrosion rates are similar to those of United States and Canadian mills when compared in terms of millimeters per thousand cooks rather than years of service. In two mills which installed spray-type recovery furnaces there was a considerable increase in corrosion rate.

Swedish work on the problem was described by Ruus and Stockman (*195*). The effect of white liquor has been studied and it has been shown that corrosion increases with increasing concentration of sulphide, hydroxide, thiosulphate, and polysulphide, but only slight corrosion by the first two in normal concentrations. It was found possible to decrease or even stop corrosion by the use of certain inhibitors, but those studied were too expensive or irregular.

Perry (*196*) described the experiences in Australia with seven kraft digesters used for kraft pulping eucalypt and pine chips. Two of the digesters were stainless steel lined, the others of C-steel. The latter corroded at 0.02 i.p.y. giving a life of 20 years, and it was felt more economical to use mild steel than stainless. The use of 5% nickel steel would increase the life four times. White (*197*) gave the results of corrosion tests made by a group of metallurgists in Australia, using various metals and liquors normally encountered in the various operations of a pulp and paper mill. The many applications of stainless steel at the new kraft mill of St. Regis at Jacksonville have been briefly listed (*198*).

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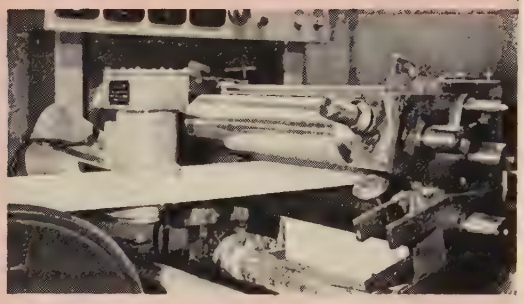

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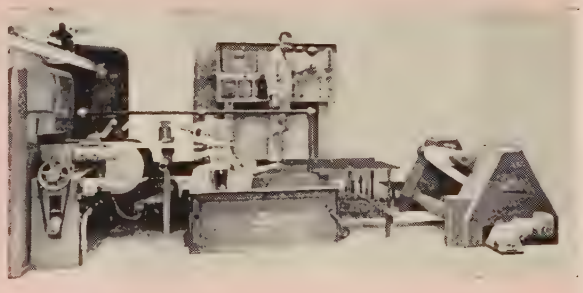

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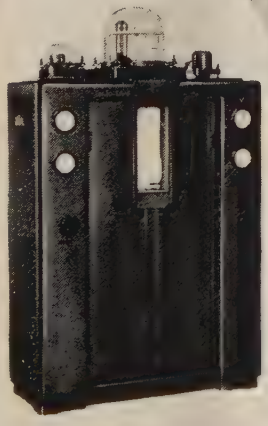

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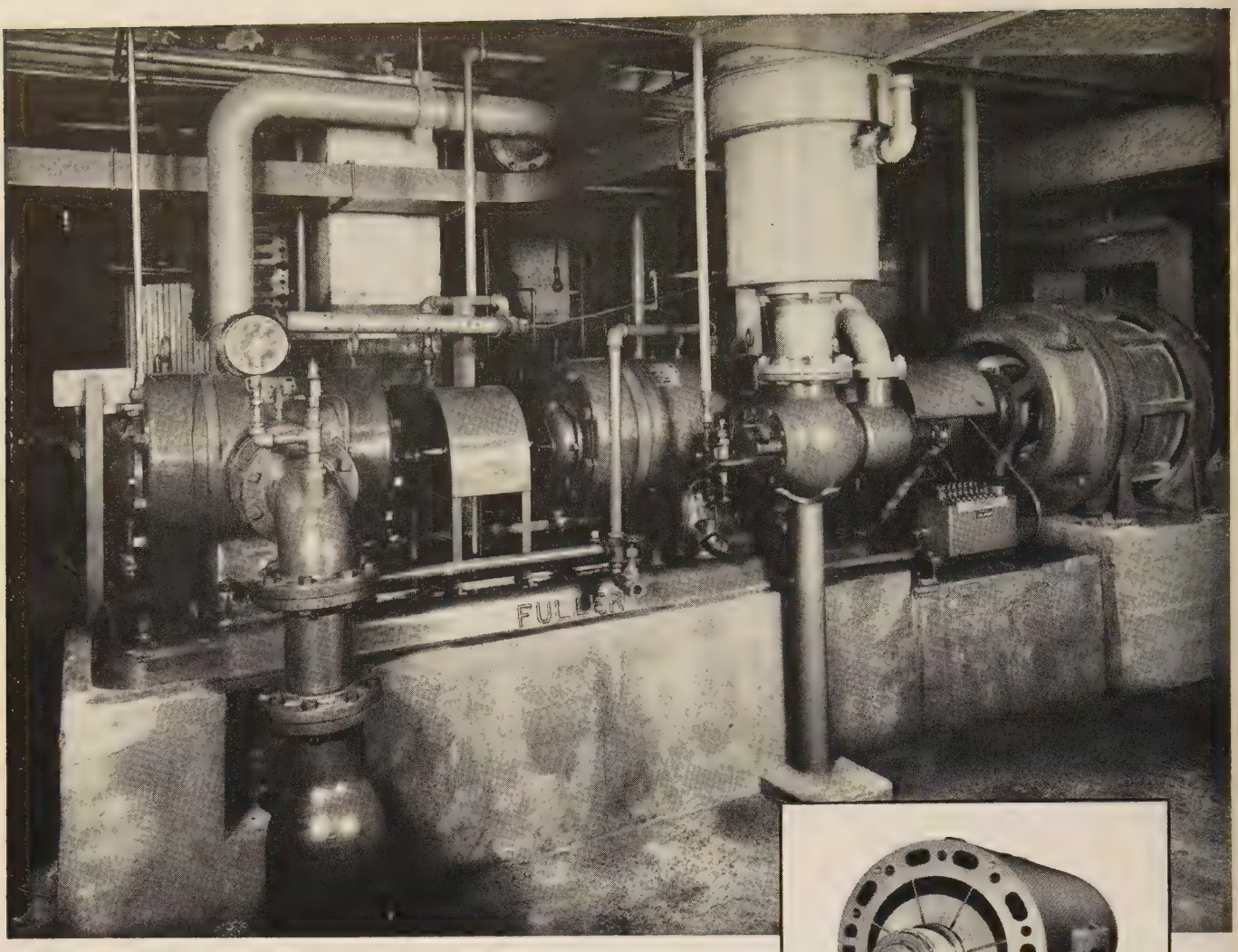


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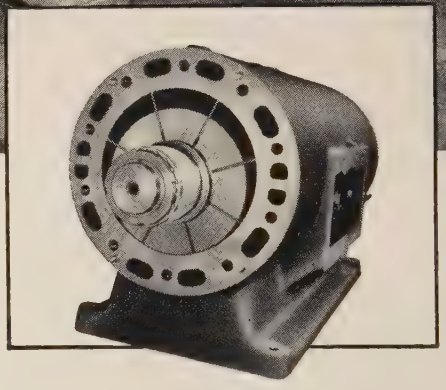

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C-232
1935

PIONEERS OF HIGH-EFFICIENCY VANE-TYPE ROTARY COMPRESSORS

*At left: Engineer's model of the Ketchikan Pulp Company's mill, now nearly completed, at Ketchikan, Alaska.*

*Alloy-lined shells for the digesters being assembled on the mill site by A.O. Smith welding crews.*

# Alaska's first pulp mill features six SMITHlined 17′ x 58′ Digesters

**Alaska's first** pulp mill has the distinction of being the largest field assembly job of welded sulphite digesters ever undertaken for a paper mill. It comprised erecting six 17-foot diameter by 58-foot high pulp digesters. Each is SMITHlined with two alloys, one to prevent corrosion by the liquor, the other to solve vapor corrosion problems in the digester domes.

**The ring sections,** heads, cones and skirts were all shipped from Milwaukee in halves, using the rails to Prince Rupert, B. C., and barges from there to Ketchikan.

**This installation** reveals the capacity of A. O. Smith to help any paper mill, large or small, to solve its digester corrosion problems. It also demonstrates the flexibility of the exclusive SMITHlining process. Two different types of alloy linings are resistance spot-welded to the carbon steel backing. In this instance, type 316L stainless is the lining in contact with the liquor, and Hastelloy alloy F, the lining in the vapor area in the dome. SMITHlining is the only commercial, automatic process for applying Hastelloy alloy F to carbon steel.

**Our metallurgical engineers** conduct exhaustive research and testing programs for the paper industry to secure reliable data on the corrosion rates of alloy materials for digester service. As a result, they were able to provide data on the alloy most suitable for resisting each type of corrosive attack in the Ketchikan digesters.

**After the digesters** have been in service, our metallurgists will make two periodic inspections with mill personnel to train them in making future inspections. This is a service we render our customers on the digesters we manufacture so that they will be able to evaluate any changes in the surface of the alloy due to process variations, and reduce maintenance to a minimum.

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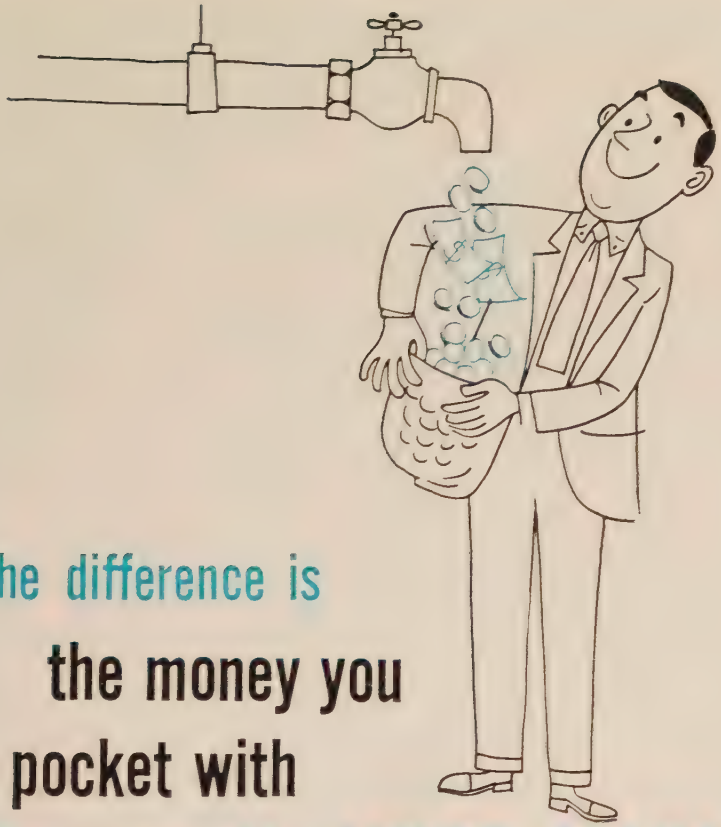

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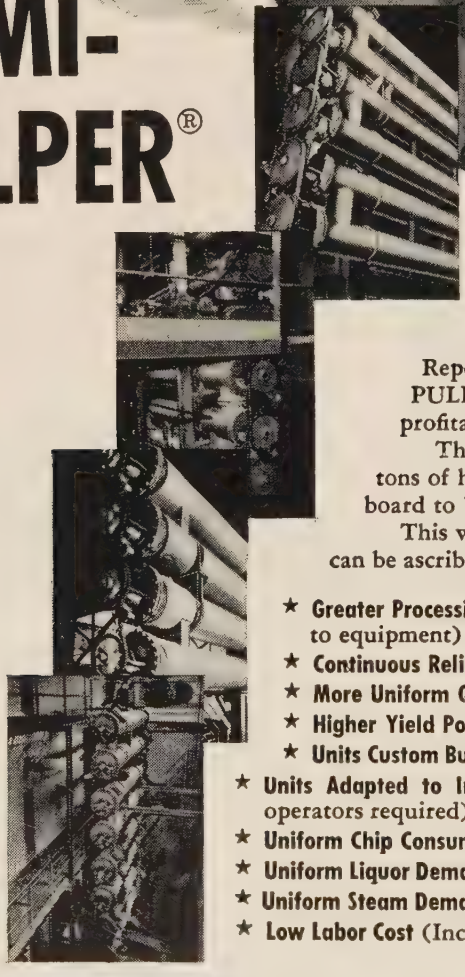

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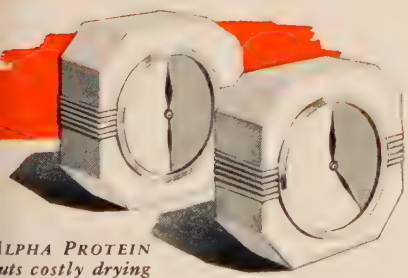

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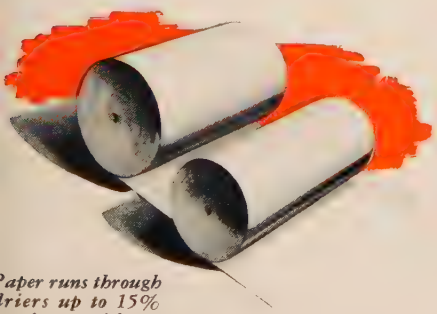

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## 1. *Relationship of Sheer Value to Pipe Friction for Bleached Sulphite Pulp Slurries*

RICHARD E. DURST and LYLE C. JENNESS

**Flow data have been taken on a long-fibered bleached sulphite stock of 2.0, 3.0, 4.0, 5.0, and 6.0% consistencies at stock velocities ranging from 1.5 to 10.0 f.p.s. in a flow system of 6-in. spiral welded pipe with test sections of 10, 20, 30, and 40-ft. lengths. Shear studies have been made on the same consistency stocks to obtain empirical shear values over the same range of velocities. A correlation of the data from the two systems has been tested for predicting friction head losses in pipes of 6, 8, 10, and 12 in. diameter with reasonable success.**

THE problems involved in the transportation through pipes of solids suspended in a fluid have been studied by workers in the fields of specialized suspensions types. Sewage sludges (*1*, *2*) have received considerable attention. Cement slurries, clay slurries, drilling muds, and grain suspensions likewise have been studied. In the pulp and paper industry the flow of pulp suspensions remains a major problem in mill design and mill operation.

The numerous sets of data compiled by various investigators (*3–9*) have been used extensively in the solution of pulp flow problems. Inconsistencies exist in the published data and the several attempts to develop correlations (*10*, *11*, *12*) have emphasized the inconsistencies. In some cases very little information was given by the investigators defining the condition under which the studies were carried out. If this had been done more fully some of the differences in the sets of data might have been resolved.

The investigation which has been carried on at the University of Maine for the past two years has had as its objective the development of a correlation which would make possible the prediction of pipe friction, using data obtained under specified conditions, and a property of the pulp stocks which could be determined by laboratory measurement. This paper presents such a correlation for a long-fibered bleached sulphite pulp.

### SUSPENSION FLOW THEORY

The behavior of true fluids in flow systems can be predicted with relative reliability due to the constancy of the fluid property, viscosity, of any such fluid. Laws and equations relating flow to the characteristics of the fluid and the system under investigation have wide acceptance.

Suspensions are characterized by the absence of a property which defines the flow behavior. Suspensions are two phase systems in which the solid, suspended phase may vary in concentration from a very small fraction of the entire mass to such a concentration that the suspending fluid approaches the limit beyond which it would no longer be the continuous phase.

The studies that have been made of two-phase systems have resulted in these systems being classified as plastics, pseudo-plastics, and dilatants or inverted plastics depending upon the nature of the shear stress-velocity diagram for the material in question (Fig. 1). It has been suggested (*11–13*) that paper pulp suspensions should be classed as plastics or pseudo-plastics.

The work of Victor P. Head (*14*) indicated that an apparent yield stress which varied with consistency was a characteristic of pulp stocks which would indicate that pulp suspensions have this property common to plastics. His work indicated the slope of the shear diagram was approximately zero.

The dimensional ratio of pulp fiber has been considered as a factor which would extend the velocity range over which laminar flow would persist (*13*), that is, the fibers would contribute to streaming within the body of the suspension. Furthermore, "slippage flow" is expected to occur at the pipe wall due to a thin film of water essentially free from fibers existing between the main body of the moving pulp and the pipe wall. Brecht and Heller (*3*) plotted profiles of the pulp flow while attempting to define the nature of the flow pattern. Their measurements tend to show "turbulent" flow starting at about 10 f.p.s. for a 3.3% stock in a 5.9-in. diameter copper pipe. Pitot tube measurements in the moving stock stream were found to be unreliable below velocities of 10 f.p.s. The principal industrial interest in pulp flow is in the velocity range of 1.5 to 10 f.p.s. This is also the range which includes the anomalies which make the prediction of pulp friction head loss difficult.

### THE EXPERIMENTAL PROGRAM

The problem of pulp stock transportation in pipes was not amenable to solution by compiling more head loss data alone. It was recognized that a property of the suspension must be evaluated which, coupled with the flow data would result in a correlation of pulp stock properties and the frictional head loss for such stocks flowing through pipes.

A property of the suspension which it is desirable to evaluate is recognized as being complex in that its value would be dependent upon several variables. These variables in turn cannot now be evaluated separately in their effect on the flow of the suspension. The known variables which make up the property of the stock include fiber length, degree of hydration or free-

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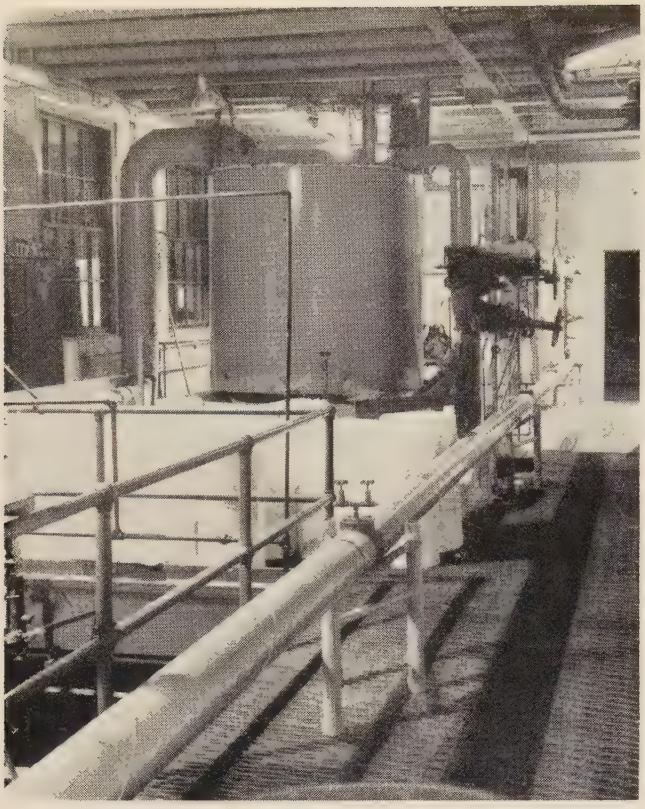


*Calibrated stock chest and 6.34-in. id. test sections*

ness, method of pulping and bleaching, temperature, and the presence of air in the stock. The pulp variables resulting from pulp source and treatment can be considered together. The combined effects of the pulp variables considered together as a property was the one chosen for study.

The temperature variable is independent and requires only the proper physical equipment to control it. The air or more generally the gaseous content of the stock is dependent upon the pulp variables and pressure and mechanical handling of the suspensions. It therefore is more difficult to control and its effect is largely unknown.

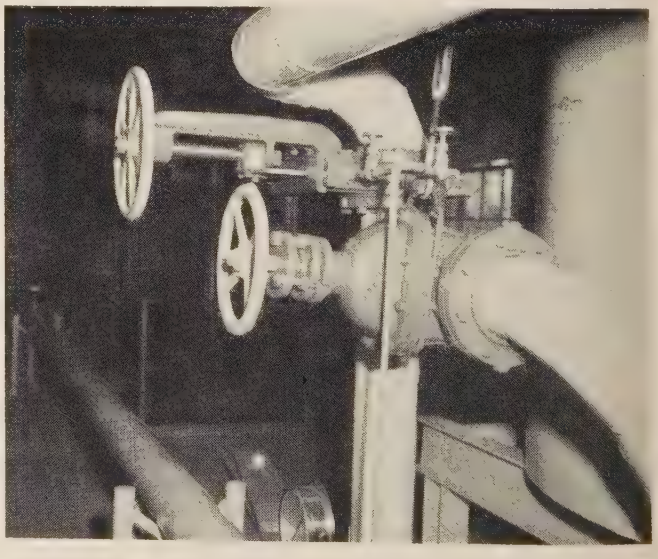
### *The Flow System*

The accomplishment of the above purpose was undertaken by the Department of Chemical Engineering at the University of Maine by constructing a pulp flow system for acquiring new data, and by building a shear tester to obtain shear data for establishing a basis for correlation. The accompanying photographs show parts of the flow system. The essential features include the stock preparation equipment, a 3-ft. Shartle Dilts Hydrapulper, and a 2600-gal. stock chest mounted on an Allis-Chalmers high-density feeder, which serves as the primary stock chest. Mounted over this chest is a second chest fitted with a quick acting door for discharging stock into the primary chest. The upper chest is calibrated for determining the volumetric flow rate of stock through the flow system. Stock is pumped through the system with an Allis-Chalmers high-density pump driven by a 75-hp. motor. The piping of the system is nominal 6-in., 6.34-in. inside diameter spiral welded steel pipe through the test section. The return section discharging into the calibrated chest is 12-in. pipe, used to reduce the total head of the system. The test sections are so arranged that pressure drops can be measured simultaneously over 10, 20, 30, or 40-ft. lengths. The shortest calming sections are 10 ft., preceding and following the 40-ft. test section. The 20-ft. test section has 20-ft. calming sections. The flow is controlled by means of a flat plate V-notch gate valve. The pressures at the test points are measured by an air-balance system using a carefully calibrated bourdon gage capable of being read in 1-oz. increments of pressure.

The air balance system is piped with $^1/_4$-in. copper tubing connected to valves at each pressure tap. The line is attached also to an air pressure tank and the bordon gage by appropriate valves. A damping chamber near the gage serves to reduce gage needle vibrations. A pressure measurement is taken at any tap by first establishing in the air line between the gage and tap a pressure greater than that existing in the flow pipes. When the air valve at the tap is opened, air escapes into the flowing stock until the pipe line pressure balances the pressure of the residual air in the air line. The pressure is indicated in the bourdon gage, and is recorded as the pressure in the flow system at the tap point being measured. The procedure for taking data of the pressures at the flow pipe test points is described in detail later.

### *The Stock Shear Tester*

The stock shear tester was designed to include the basic scheme proposed by Victor Head (*14*); that is, a stock container and brush rotor of large dimensions compared to the length of the pulp fibers being studied. The stock cup is 15.624 in. inside diameter and 18.0 in. inside height. The cup is suspended on special ball bearings and is free to rotate through an arc of 15°. The cup support was designed to reduce the friction as much as possible with the result that a force of 5 grams will rotate the cup. The brush rotor was made by bolting four nominal 10-in. wire buffing wheels into a unit with a diameter of 9.875 in. and 7.5 in. in height. The ends are of smooth aluminum plate to reduce end effects as much as possible. The location of the rotor with respect to the cup is indicated by the following dimensions:



*Stock flow regulating valve*

| | |
|---|---|
| Lower end of rotor to bottom of cup | 4.25 in. |
| Upper end of rotor to top of cup | 6.25 in. |
| Rotor peripheral surface to cup wall | 2.87 in. |

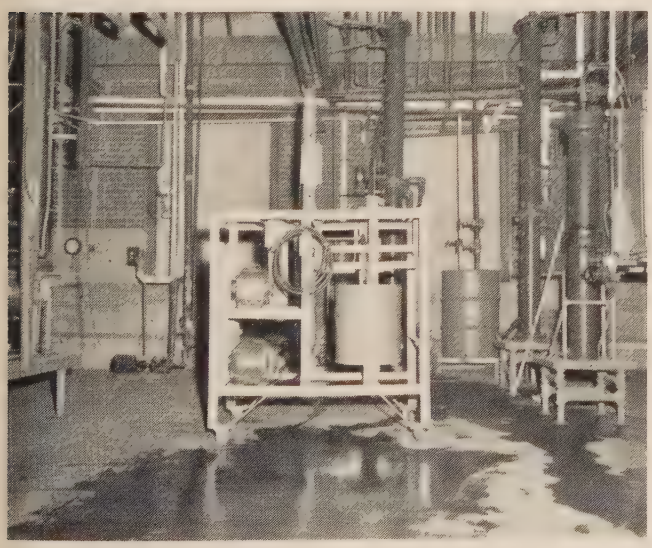
The rotor is capable of being driven at positive speeds ranging from 28 to 600 r.p.m. by means of a 1-hp. motor acting through a Link-Belt P.I.V. unit and the appropriate pinions with silent chain drive. The force applied to the suspended cup through the stock under test by the rotor is measured by a spring balance attached by means of a light cable to a point directly above the cup edge. Provision is made for maintaining the cable attachment from the scale to the cup support tangential to the cup at all times.

## FRICTION LOSS DETERMINATIONS AND RESULTS

The stock selected for study in the flow system and the shear tester consisted of bleached sulphite pulp of 0.90 mm. average fiber length and Canadian Standard freeness of about 650. This stock was chosen due to the similarity of its properties with those of the sulphite stock used by Brecht and Heller (*3*). The stock, received as dry lap was made into a slurry of the required consistency using the hydrapulper and the high-density feeder. The pulp stocks prepared in this manner were well dispersed and free of pulp masses which would have contributed to a nonuniform stock.

The procedure for taking pressure drop data was carefully standardized to eliminate errors or difference due to irregular operating techniques. It was believed necessary to take the pressure drop data at increments of stock velocity of 0.5 ft. per sec. beginning at 1.5 ft. and continuing up to 10 f.p.s. The mass flow rate of the stock was determined by timing the interval necessary to fill the calibrated stock chest to a predetermined depth. The measured volume was 127 cu. ft. which would be filled in 57 sec. at a flow rate of 10 f.p.s. through the 6-in. test system. The volume of stock was sufficient to give timing intervals sufficiently long to reduce small differences between rate determinations of no appreciable significance. A flow rate was acceptable when the difference between two determinations was not more than 1%.

The air balance system of reading pressures at the test points made it possible to read all values from a single gage. One operator was assigned the responsibility of reading the pressure and recording the data. A total of 36 readings were taken, nine at each pressure test point, for each stock velocity. After 24 readings were taken the flow rate was checked to assure uniformity, the 12 remaining pressure readings then being taken for that velocity. Table I shows the order of taking the pressure data. It was believed this manner of taking the data should expose any irregularities that might occur during a test. The pressure at each test point used in calculations was the average of the nine readings taken. The pressure drop between any two test points was converted to the frictional head, expressed in feet of water for a 100-ft. length of pipe. The results of the flow study on bleached sulphite stocks of 2.0, 3.0, 4.0, 5.0, and 6.0% consistency at temperatures between 84 and 88°F. are given in Fig. 2.



*The stock shear tester*

**Nomenclature**

| *Symbol* | *Definition* | *Units* |
|---|---|---|
| $\Delta H_f$ | Head loss due to friction | Feet |
| $D$ | Internal diameter of pipe | Feet |
| $u$ | Average velocity of stock flowing | Feet per second |
| $L$ | Length of pipe | Feet |
| $C$ | Consistency of stock | Mass of moisture-free stock/mass of suspension |
| $\rho$ | Density of stock | Slugs per cubic foot |
| $f$ | Friction factor $\Delta H_f D 2gc/u^2 L$ | Dimensionless |
| $N_{SF}$ | $Du\rho/s$, suspension flow number | Dimensionless |
| $S$ | Shear value | (Pounds force × seconds)/$L^2$ |

## OPERATIONS OF THE SHEAR TESTER AND RESULTS

The shear value of the several consistency stocks has been determined using the shear tester previously described. Considerable exploratory and developmental work was necessary before an acceptable method of operating the tester was established. The standard procedure for operating the shear tester in testing the pulp stocks studied specifies that the cup be filled to the upper end of the rotor with the stock. The rotor turning at any selected speed sets up forces which tend to rotate the stock cup. The cup is restrained by the spring balance which measures the force applied to the cup. The shear value of a stock as now determined varies with the peripheral speed of the rotor. It was learned that in order to obtain reproducible data, equilibrium conditions must be established within the stock. Stock formation has been found to change to a marked degree as the rotor speed varies. The lower consistency stocks will come to equilibrium in relatively short time as indicated by the constancy of the measured force. As the consistency increases the time required for steady-state readings to be attained increases rapidly. It is possible to see the changes in stock formation as the rotor speed is varied. The changes in stock formation as observed in the shear tester are believed to be an important factor in the friction loss of the stock flowing in pipes, however, it has not been possible to explore the relationship up to this time.

The force recorded at steady state for a given rotor speed provided the data for computing the stock shear value at that condition. The shear value as used here is the force in pounds divided by the rotor peripheral

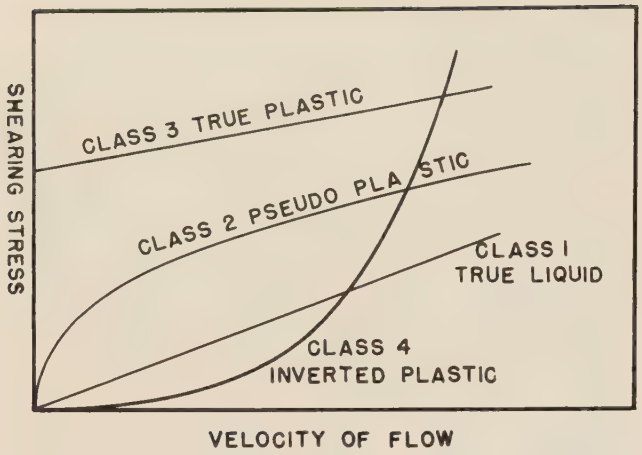



*Fig. 1. Flow characteristics of type materials*

area in square feet per second; in dimensional units,

$$s = \frac{FT}{L^2}$$

The area term is fully defined by the product of the peripheral area of the rotor times the rotor revolutions per second. The shear value of the stocks has been plotted as the ordinate with the rotor peripheral velocity in feet per second as the abscissa on fully logarithmic paper, Fig. 3.

## CORRELATION OF FRICTION DATA WITH SHEAR VALUES

The determined shear values made possible a correlation of the friction loss data with a stock property that could be measured in a laboratory instrument. The shear value with the dimensions $FT/L^2$ when combined with the pipe diameter, stock velocity, and stock density in slugs per cubic foot forms a dimensionless group which we propose as a Suspension Flow Number,

$$N_{SF} = \frac{Du\rho}{s} \qquad (1)$$

which may have the same significance in suspension flow the Reynolds number has in the flow of true fluids. It is only necessary then to write the expression

$$\frac{\Delta H_f D 2gc}{u^2 L} = f = \phi \frac{Du\rho}{s} \qquad (2)$$

compute the value of the friction factor, $f$, for observed values of the friction loss at selected stock velocities through the pipe of known diameter and length. The $N_{SF}$ may be computed by substituting measured shear values at the same rotor velocities as the stock velocities used in the computation of the friction factor. The data points obtained by such computation from the flow and shear data for the sulphite stock studied appear in Fig. 4. The straight line appearing on the chart is the averaged representation of the data points for the five consistencies of stock investigated.

The proposed correlation is empirical since the shear value used in the $N_{SF}$ is a function of the shear tester rotor peripheral velocity and is not a measured stock velocity.

## APPLICATION OF THE CORRELATION

The correlation has been tested for predicting the friction head loss of sulphite stock for other pipe diameters than the 6 in. by comparing the calculated friction head loss with that reported by other investigators. Such testing is possible only by assuming the shear value of the stock used in obtaining the earlier data was the same as that used in this investigation.

The first body of friction data used was the Forrest and Grierson data for sulphite pulp stock flowing through cast iron pipe of 6, 8, 10, and 12 in. diameter. Considering the difference in the pipe used in the two studies the proposed correlation predicted friction loss values an average of 68.4% lower than the friction loss observed by Forrest and Grierson for stocks of 3, 4, and 5% consistency flowing in 6-in. pipe, and for 2, 3, 4, and 5% consistencies flowing in the 8, 10, and 12-in. pipes. A total of 38 comparisons were made, with the minimum deviation being 38.3% and the maximum being 88%. Further analysis showed that 44.7% of the comparisons were within ±10% of the mean deviation and 71% of the comparisons were within ±20% of the mean.

A similar comparison was made using the data of Brecht and Heller. A total of 50 comparison calculations were made for stock flowing in copper pipe of 5.9 and 7.87 in. diameter in the velocity range of 2 to 10 f.p.s. The average deviation of the calculated head loss was 22.3% lower than the observed when compared to the observed values.

Comparison of the friction curves for the two sets of data shows a remarkable similarity exists. However, it should be observed that the maximum and minimum points in the University of Maine curves occur at lower stock velocities for a given consistency than in

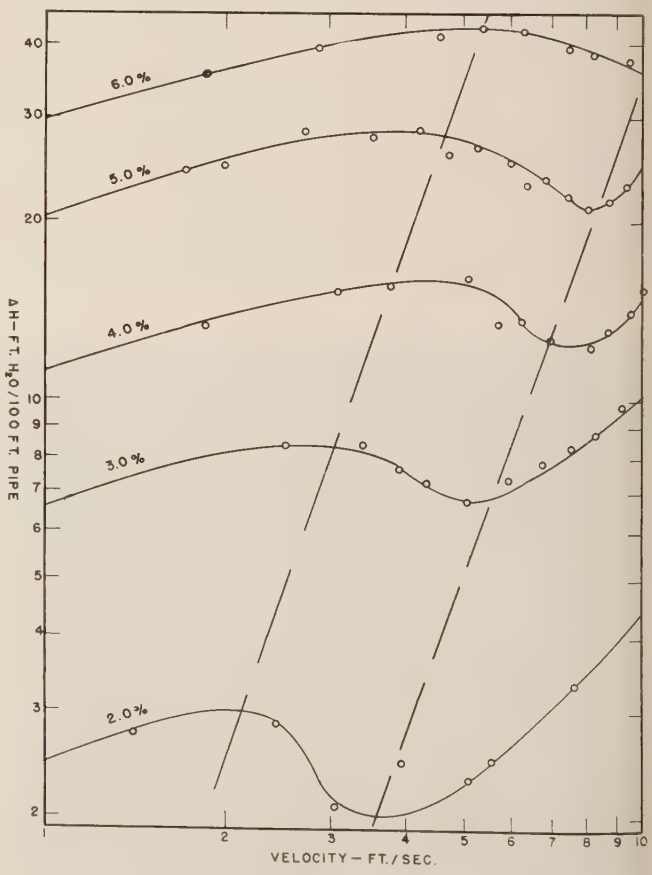



*Fig. 2. Pressure drop—velocity relationships for bleached sulphite stock through 6.34-in. spiral-welded steel pipe*

the Brecht and Heller curves. Some very limited data tend to indicate the location of the maxima and minima of the curves with respect to stock velocity is a function of the length of the calming sections preceding the test section. That is, as the calming section increases in length the maxima and minima tend to be displaced to the left, occurring at lower stock velocities than when short calming sections are employed. In this investigation the calming sections could be varied from 10 to 40 ft. in length. The calming sections in the Brecht and Heller flow system were not given; however from the description of the equipment it appears that some straight sections preceding test points were not more than a few pipe diameters in length.

## DISCUSSION

It was desirable to fix as many of the known variables as possible during the testing program. As indicated the stock consistency and flow velocity could be controlled within desired limits. The stock temperature was found to be a more difficult variable to control. Some control of the temperature rise which accompanied continuous pumping was possible by keeping the 12-in. diameter pipe section covered with a film of cooling water. Since isothermal conditions could not be established it was decided to operate over a temperature range which could be maintained. This temperature range was found to be 84 to 88°F. Over this range the effect of temperature is believed to be negligible.

The air content of the pulp suspension has been a cause of concern in this study. Limited investigation has indicated the percentage of adsorbed air on the pulp fibers can become appreciable. The amount of air which occurs as free masses within the suspension was found to be less than 1% by volume. The free air in the amount found is believed to be of little importance but the amount of adsorbed air apparently can vary to a large degree and its effect is now unknown.

Observations made on the stock under test in the shear tester have indicated that stock formation has

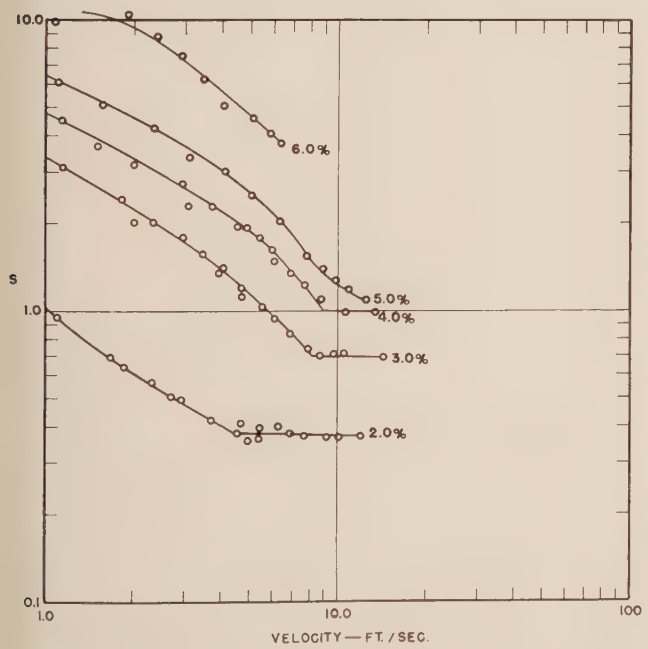



*Fig. 3. Shear value versus rotor velocity for bleached sulphite stock*

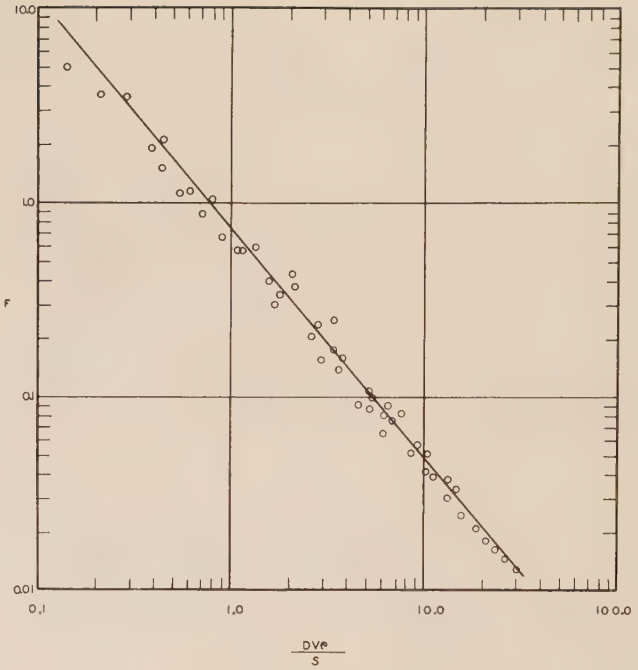



*Fig. 4. Friction factor versus suspension flow number, bleached sulphite stock*

an important bearing on the frictional loss of pulps flowing in pipes. A year ago it was proposed that the rapid increase in the friction with increasing stock velocity, up to the first maximum in the head loss curve could be explained by the presence of stationary stock in the pipe line. Movement of stock over the stationary mass would result in higher friction since stock was shearing over stock. Furthermore, the stagnant stock reduced the effective cross-sectional area of the pipe. The reduced pipe size then necessitates a higher velocity in the moving stock than was calculated assuming the entire cross section of the pipe was available for transporting stock. The unpredictable pitot tube readings reported by Brecht and Heller for velocities below 10 f.p.s. and some exploratory work during the past Summer tend to confirm the presence of "dead" spots in the stock mass. These spots have been found to consist of stationary stock nearly 1 in. thick on the lower half of the 6-in. pipe when pumping 4% stock at velocities below 4 f.p.s.

## CONCLUSIONS

The results obtained on a bleached sulphite pulp stock of consistencies from 2 to 6% tend to agree with the Brecht and Heller data considering the differences

**Table I. Typical Flow Data**

| *Flow time* | $P_1$ | $P_2$ | $P_3$ | $P_4$ |
|---|---|---|---|---|
| 60.6 | 11–13 | 10–12 | 8–13 | 7–14 |
| 60.8 | 11–13 | 10–12 | .. | .. |
| | ... | 10–12 | 8–12 | .. |
| | . | ... | 8–13 | 7–13 |
| | 11–12 | ... | .. | 7–14 |
| | 11–12 | 10–12 | 8–12 | 7–12 |
| | 11–12 | 10–11 | .. | .. |
| | ... | 10–11 | 8–12 | .. |
| | ... | ... | 8–12 | 7–12 |
| | 11–12 | ... | .. | 7–12 |
| 60.4 | 11–13 | 10–12 | 8–13 | 7–13 |
| | 11–12 | 10–12 | .. | .. |
| | . . | 10–12 | 8–13 | .. |
| | .. | ... | 8–13 | 7–13 |
| | 11–12 | ... | .. | 7–13 |

in the flow systems. There is some evidence that the length of the calming sections has some effect on maxima and minima in the head loss curves.

A property of pulp suspensions has been measured in the shear tester and expressed in empirical units as the shear value. The shear value combined with the stock velocity, pipe diameter, and pulp density form a dimensionless group which has been designated the Suspension Flow number, $N_{SF}$. The $N_{SF}$ combined with a friction factor computed from measured friction head loss data gives a friction factor plot which is useful for predicting head loss in pipes carrying pulp stock suspensions. The application is restricted at this time to pulps having properties similar to those of the pulp used in the investigation.

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# Note on the Chemistry of the Cambial Zone of Black Spruce


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**A summative proximate analysis of the cambial zone of black spruce showed that the dry material was very high in extractives which contained much sucrose. The cambium also contained considerable protein, appreciable amounts of pectic material, but very little lignin and relatively small amounts of cellulose and hemicelluloses. That fraction of the zone which was most intensively studied, and which represented about 20% of its total dry weight, contained the cellulose and most of the initial lignin; it also retained an appreciable portion of the initial protein. The lignin/cellulose ratio in the cambium was much lower than that found even in young sprucewood. Studies of the cambium of various woods are being continued at The Institute of Paper Chemistry.**


In 1947, Anderson and Pigman (*1*) reported briefly on a preliminary chemical study of the inner bark and the cambial zone of a black spruce. The present paper gives a résumé of work which supplemented their investigations, and which may have some bearing on the studies that are now being conducted intensively by another group at The Institute of Paper Chemistry. Our work was carried out 8 years ago, prior to the advent of chromatographic techniques, and none of the materials then studied are now at hand. However, even these fragmentary data appear to merit brief publication at this time, though future refinements will undoubtedly be made.

The log from a tree about 65 years old was felled on June 3, 1946, and cut into eight bolts. The outer bark was rubbed off with a draw-knife and carried with it a portion of the inner bark. The remainder of the inner bark was then peeled off, and subsequently the moist cambial layer scraped off from the young wood with the back of a knife. It may be pointed out again that this layer or zone contains the protoplasm which takes part in those life processes giving rise to the synthesis of woody tissue and the bark of the tree.

As the removal of this moist, colloidal mass C was by no means a clean-cut separation, it undoubtedly contained small amounts of the adjacent, recently formed sapwood. A portion of C, termed $C_1$, was dried rapidly in a current of warm air, followed by further drying *in vacuo*. This amounted to about 61 grams. Another portion, $C_2$, was extracted immediately with boiling alcohol, and after drying, the alcohol-insoluble fraction of $C_2$ weighed 13 grams. The alcohol extract weighed 31.5 grams, and hence the original dry $C_2$ amounted to about 44.5 grams.

$C_1$ was extracted successively with benzene and with 95% alcohol, yielding 6.75 and 60.55% extractives, respectively. This benzene extract of $C_1$ gave an unexplored yellow oil together with small amounts of a white solid (which had been flushed out of the cambium rather than dissolved in the solvent). The latter appeared to be a color precursor similar to material isolated from the inner bark of spruce which formed the basis of another publication (*2*). A portion of $C_1$, which was insoluble in benzene and alcohol, was combined with a part of the insoluble portion of $C_2$, and this combined sample (22 grams) was triturated within a mortar with cold 1% hydrochloric acid, followed by the addition of 7 volumes of alcohol, thus yielding a gelatinous material which was then treated with 5%


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ammonium hydroxide. The resulting insoluble slimy fraction $C_3$ was centrifuged and filtered, and the filtrate treated with acetic acid and aqueous calcium chloride, thus yielding, after washing with aqueous alcohol $C_4$, an impure calcium pectate, contaminated with appreciable amounts of protein. This contamination was evident from the analysis which indicated 55.5% uronic anhydride, 33.5% pentosan, and 2.84% nitrogen. The filtrate from $C_4$, when treated with a large excess of alcohol, gave a relatively small amount of precipitate which was largely proteinaceous, but which carried a significant amount of polysaccharidic material. It contained 4.72% nitrogen (equivalent to about 29.5% protein), 12.8% uronic anhydride, and 12.8% pentosan.

$C_3$ (insoluble in aqueous ammonia) was then macerated with 3% sodium hydroxide, allowed to stand 3 hr., filtered through cloth (and refiltered until clear), and washed with water. The alkaline filtrate and washings were acidified with acetic acid and treated with 6 volumes of alcohol. The resulting precipitate ($C_5$) was also a mixture of protein and polysaccharides and contained 2.48% nitrogen (equivalent to 17.5% protein), 15.6% uronic anhydride, 37.2% pentosans, and 1.07% methoxyl. The brown fibrous residue (termed $C_6$) which was *insoluble* in 3% alkali formed the starting material of the present study. It represented approximately 20% of the dry weight of the original unextracted cambial zone (i.e., $C_1 + C_2$).

## STUDY OF CAMBIAL ZONE RESIDUE ($C_6$)

$C_6$ gave the following analytical values: ash—0.54%, nitrogen—2.48% (equivalent to 15.5% protein), uronic anhydride—6.04%, pentosans—8.38%, and methoxyl—1.87%.

### *Lignin*

When subjected to the usual Klason lignin determination, three samples of the cambial zone residue $C_6$ gave (a) 12.9, (b) 13.1, and (c) 12.3% of "apparent lignin." The mixture of the lignins obtained in the first two determinations (a + b) contained 4.21% nitrogen and 8.83% methoxyl; the third lignin preparation contained 4.23% nitrogen and 9.7% methoxyl.

It is evident that the high protein (nitrogen) content of the cambial zone residue interfered seriously with the lignin determination, probably through a coprecipitation of a sugar-amino acid complex (melanoidin reaction), which was not reproducible. Therefore, the lignin determinations gave variable and fictitiously high results. Furthermore, it is impossible to correct these results by subtracting the apparent protein content ($4.22 \times 6.25$), because sugar is also involved in the precipitation.

Previous work has shown that the lignin of spruce heartwood contained 14.95% methoxyl; that the lignin from spruce sapwood contained 14.85 to 15.0% methoxyl; and that lignin from the youngest sapwood contained 14.15 to 14.95% methoxyl. If the hypothesis is ventured that the "true" lignin of the cambial zone residue also contains approximately 14.8% methoxyl, the following calculations may be made.

The percentage of methoxyl in the uncorrected lignin (a + b), calculated on the basis of the original cambial zone residue, is 1.16%. This, multiplied by 6.83 (the factor for converting methoxyl to lignin (gives 7.9% as the "true" lignin content of the cambial zone residue. Similarly, the methoxyl content of the (c) preparation, on the basis of the original cambial zone residue, is 1.19%, which gives 8.1% as the "true" lignin content of the cambial zone residue. It is obvious that this calculation is speculative.

### *Holocellulose*

By the usual chloriting procedure (*3*) but using only one chlorite treatment, a yield of 88.9% of crude "holocellulose" was obtained. This holocellulose still retained 1.30% methoxyl and 1.04% nitrogen. Inasmuch as the lignin methoxyl (based on the cambial zone residue) was 1.18%, and since the total methoxyl in the residue was 1.87%, the expectancy of methoxyl in the holocellulose based on this residue would be 1.87 minus 1.18 or 0.69%, whereas it is actually $0.889 \times 1.30$ or 1.16%. The difference (0.47%) presumably stems from the presence of lignin, which may thus be present to the extent of 3.21% (i.e., $0.47 \times 6.83$). The protein content ($1.04 \times 0.889 \times 6.25$) = 5.81%. The holocellulose yield, corrected for both lignin and protein, is thus 79.9%. Presumably this result is still high, inasmuch as experience has shown that a satisfactory lignin correction can never be applied to a chlorite holocellulose.

### *Hemicelluloses*

Using the usual technique (*4*)—i.e., by extracting the holocellulose first with 5% potassium hydroxide (A) and then with 16% potassium hydroxide (B) (under purified nitrogen)—the following results were obtained (based on the holocellulose):

| | *Ash-free yield, %* | *Nitrogen, %* | *Uronic anhydride, %* |
|---|---|---|---|
| Hemicellulose A | 14.9 | 6.2 | 9.2 |
| Hemicellulose B | 7.1 | 0.0 | 7.5 |

Unfortunately, no methoxyl determinations were made but, as shown below, the excess methoxyl must have been associated largely with the alpha-cellulose (i.e., the residue from the above extractions with alkali). Furthermore, inasmuch as the alkaline solutions of the hemicelluloses were nearly colorless, presumably little lignin could have been present. On the other hand, the 5% potassium hydroxide solution frothed greatly and had an atypical odor, indicating the possible presence of protein. The 16% potassium hydroxide solution, however, showed no such property. After correcting hemicellulose A for protein, calculation shows that the hemicellulose A content of the original cambial zone residue is 8.08%—i.e., $[14.9 - (14.9 \times 0.062 \times 6.25) \times 0.889]$. The hemicellulose B content of the original cambial zone residue is calculated as 6.3% (i.e., $7.1 \times 0.889$).

### *Alpha-Cellulose*

The acid-washed residue left after extraction of the holocellulose with potassium hydroxide amounted to about 70% (based on the holocellulose). However, this alpha-cellulose fraction contained 0.15% nitrogen and 0.51% methoxyl, indicating retention of both lignin and protein. The alpha-cellulose also contained 12.9% mannan.

A very interesting experiment involved the Klason treatment of the alpha-cellulose fraction with 72%

**Table I. Composition of $C_6$[a]**

| | % |
|---|---|
| Ash | 0.54 |
| Lignin | 8.0 |
| Hemicellulose A | 8.1 |
| Hemicellulose B | 6.3 |
| Alpha-cellulose | 59.5 |
| Glucan in cellulose | 51.8 |
| Mannan in cellulose | 7.7 |
| Protein (2.48% N × 6.25) | 15.5 |
| Total | 97.94 |

[a] Based on oven-dry weight of $C_6$.

sulphuric acid, followed by the usual 3% hydrolysis. In one case (I) 30 ml. of sulphuric acid were used for 2 grams of alpha-cellulose; in the other case (II) 35 ml. of sulphuric acid were used. In the second case, the mixture was accidentally diluted with water, and therefore the results are not strictly comparable.

In either case, the hydrolysis yielded an atypical, abnormally large flocculent precipitate resembling lignin. Evidently it was lignin contaminated in (I) with a melanoidin condensation product, and in (II) with both a melanoidin-like product and probably an incompletely hydrolyzed polysaccharide (hydrocellulose) resulting from the dilution of the 72% acid. These conclusions are drawn from the following data on the "lignin like" residue:

| | *Yield (based on alpha-cellulose), %* | *Nitrogen, %* | *MeO, %* |
|---|---|---|---|
| I | 4.2 | 0.53 | 11.0 |
| II | 8.9 | 0.03 | 5.4 |

In other words, the higher the yield of this impure "lignin," the lower its methoxyl and its nitrogen content.

If, again, we assume that the residual lignin in the alpha-cellulose contained 14.8% methoxyl and that the conversion factor is 6.83, the actual percentage of lignin [calculated from (I)] is (0.11 × 4.2 × 6.83) or 3.16, and the calculated methoxyl content of the alpha-cellulose would be 0.46% (which is in fair agreement with 0.51% methoxyl actually found).

A similar calculation for (II) gives 0.49% methoxyl in the alpha-cellulose and a "true lignin content" of 3.37%. Thus, despite the discrepancy in the amounts of (I) and (II), the calculated "methoxyl" and "lignin" values are in fairly good agreement, when recalculated on the basis of the alpha-cellulose fraction. [From the methoxyl content of the alpha-cellulose (0.51%), a lignin value of 3.48% is obtained by multiplying 0.51 × 6.83.]

**Table II. Approximate Composition of the "Cambial Zone" of Black Spruce (Oven-Dry Basis)**

| | % | |
|---|---|---|
| Fixed oils | 6.25 | |
| Leucoanthocyanin (?) | 0.50 | Total extractives |
| Sucrose | 32.50 | 67.3% |
| Other alcohol-soluble extractives | 28.05 | |
| Ash | 3.60 | |
| Protein | 7.00 | |
| Alpha-cellulose | 11.90 | |
| Lignin | 1.83 | |
| Pectic material* | 3.00 | |
| Hemicelluloses | 2.90 | |
| Other polysaccharides removed by 5% $NH_4OH$ and 3% NaOH* | 2.00 | |
| Total | 99.53 | |

**Table III. Analyses of the Cambium of Scots Pine (Oven-Dry, Unextracted Basis)**

(Data of Allsopp and Misra)

| | % |
|---|---|
| Total alcohol extractives (including 27.2% sucrose) | 62.00 |
| Cellulose (Norman and Jenkins Method) | 9.53 |
| Lignin (Klason) | 3.25 |
| Pectin | 6.30 |
| Protein | 7.90 |
| Ash | 6.11 |

If the alpha-cellulose fraction is corrected for lignin and protein [3.48 + (0.15 × 6.25)] or 4.42%, the corrected value becomes 70 minus (70 × 0.0442), or 66.9% (on the basis of the holocellulose) or 59.5% [on the basis of the original cambial zone residue $C_6$].

Table I gives the composition of the cambial zone residue $C_6$ summarizing the analytical data referred to above after applying the various corrections.

$C_6$ also yielded 1.8% acetyl, but, owing to the prior use of acetic acid this may be an artifact, and is therefore not included in the summary.

The average number degree of polymerization of the alpha-cellulose was 1085.

As indicated above, $C_6$ includes only about 20% of the dry weight of the original cambial zone. On the basis of this and of other analyses, it is possible to give an estimate of the composition of the original cambial zone. Admittedly this is a rough approximation. The values with asterisks represent rough estimates that are probably minimal values. Some of the extractives removed by alcohol were not identified, although they were known to contain simple sugars. The seeming accuracy of the summative results is purely fortuitous.

The results in Table II are in general agreement with those reported by Allsopp and Misra (*5*) for Scots pine. These (recalculated on the extract-containing basis) are given in part in Table III. No attempt has here been made to compute the hemicelluloses (from their various figures for uronic anhydride, furfural, etc.).

The analytical data of Anderson and Pigman and those of Allsopp and Misra on young sapwood (which are not reproduced here) clearly show the marked differences between the cambial zone on the one hand and newly formed wood on the other.

As in the case of spruce cambium, pine cambium shows a very high amount of alcohol-soluble material, an appreciable part of which is sucrose. In either case the protein content is high, the pectin content is appreciable, and the lignin/cellulose ratio is far lower than that in the respective woods.

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Grateful acknowledgment is made to Robert Buxton, who made many of the analyses and to William Shockley who determined the degree of polymerization of the cellulose.

# Evaluation of the SFMC-TAPPI Drainage-Time Tester

C. E. HRUBESKY and J. J. PEROT, JR.

**Two drainage-time testers manufactured to meet requirements described in TAPPI Standard T 1002 sm-51 give comparable results on a wide range of insulating board stocks if the pulps are given identical treatment. Stirring pulps with a high-speed, propeller-type stirrer alters some pulps sufficiently to give different drainage rates with different times of stirring.**

THE results of freeness tests made at the Forest Products Laboratory with several freeness testers on board stocks were reported at the February, 1948 and 1949, meetings of the Technical Association of the Pulp and Paper Industry. The Schopper-Riegler (regular), Schopper-Riegler (modified), Canadian Standard, Oliver Fir-Tex, and Williams Precision testers were compared with free, normal, and slow stocks supplied by seven board manufacturers.

The results of these tests indicated that a tester of the Williams type, modified to hold a larger specimen, would be the most suitable type for evaluating the drainage rate of coarse-fibered pulps. Accordingly, TAPPI method T 1002 sm-51 was written and specifications for a tester were drawn up by Subcommittee 4 of the Structural Fibrous Materials Committee. Two testers were manufactured. One was sent to the Flintkote Research Laboratory and the other to the Forest Products Laboratory for comparative tests to determine the reproducibility of results.

The purpose of this paper is to describe some results obtained at these two laboratories with the two testers on pulps with fast, normal, and slow drainage rates.

## EQUIPMENT

The SFMC tester, as described in TAPPI method T 1002 sm-51, consists of a glass cylinder 3 in. in diameter and approximately 19 in. long fitted into an adaptor sleeve. This sleeve slides down over the outside surface of a brass base in which is recessed a 40 by 40-mesh brass screen with an effective screen area of 7.1 sq. in. (3 in. diameter). A 2-in. drainage pipe, 36 in. long, attached to the bottom of the base serves as a water leg that is actuated by a trap and quick-opening valve connected to its bottom end.

As described in the method of test, the tester is used by measuring the time in seconds required for water to drain through the screen from 1800 ml. of a definite pulp suspension. The marks on the glass cylinders on the two testers received from the manufacturer indicated volumes of 1872 and 1874 ml., respectively.

The test is made with 10.6 grams (oven-dry equivalent) of pulp, equivalent to 1.5 grams per sq. in. of screen area, at a temperature of 80°F. (26.7°C.). The free-water value for both testers is less than 1.5 sec.

The first SFMC testers used in this work were equipped with a trap in which an air pocket formed when the cylinder was filled with water or a pulp suspension. To eliminate the air pocket, a vent was established by connecting a piece of copper tubing between the trap and the lower part of the water leg. Although the instruments equipped in this manner gave excellent reproducibility of results, it was later discovered that, with slow pulps, the vacuum in the water leg broke at a definite point before the draining stock reached the lower mark on the cylinder.

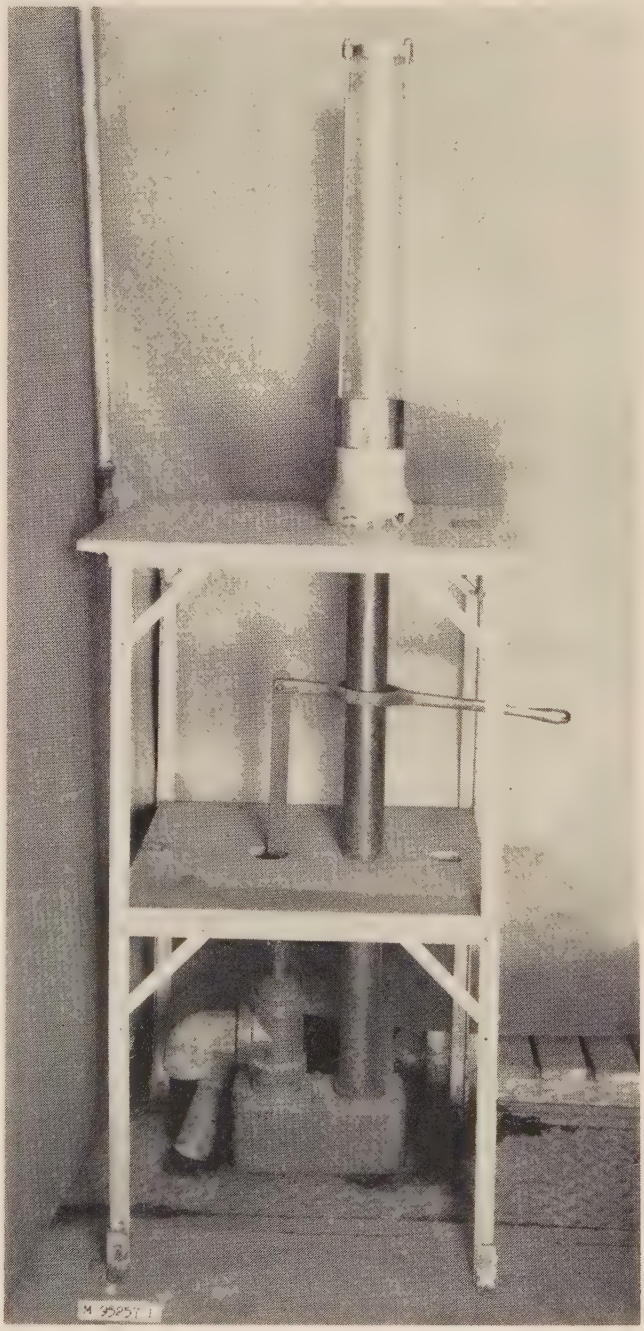

*Fig. 1. SFMC drainage-time tester*

C. E. HRUBESKY, Chemist, Forest Products Laboratory maintained at Madison, Wis., in cooperation with the University of Wisconsin, Forest Service, U. S. Department of Agriculture, and J. J. PEROT, JR., Chemist, The Flintkote Co., Whippany, N. J.

Table I. Comparison of Drainage Tests Made at the Flintkote Laboratory and at the Forest Products Laboratory

| Company | Pulp type | Average drainage time: Flintkote Laboratory, sec. | Average drainage time: Forest Products Laboratory (operator no. 1), sec. | Average drainage time: Forest Products Laboratory (operator no. 2), sec. | Deviation from the averages of the values reported, % |
|---|---|---|---|---|---|
| Celotex Corp., Marrero, La. | Fast | 17.5 | 15.0 | ... | 7.7 |
| | Normal | 34.0 | 33.0 | ... | 1.5 |
| | Slow | 64.5 | 60.0 | 56.5 | 7.0 |
| Fir-Tex Insulating Board Co., St. Helens, Ore. | Fast | 8.5 | 8.5 | ... | 0 |
| | Normal | 13.5 | 12.6 | ... | 3.8 |
| | Slow | 14.0 | 13.0 | 15.0 | 7.1 |
| Flintkote Co., Meridian, Miss. | Fast | 5.0 | 4.5 | ... | 5.3 |
| | Normal | 47.5 | 45.5 | ... | 2.2 |
| | Slow | 174.5 | 168.0 | 166.0 | 2.9 |
| Johns-Manville Corp., Manville, N. J. | Fast | 21.5 | 19.5 | ... | 4.9 |
| | Normal | 30.5 | 29.5 | ... | 1.7 |
| | Slow | 44.5 | 48.0 | 53.0 | 9.3 |
| Minnesota and Ontario Paper Co., International Falls, Minn. | Fast | 14.5 | 13.5 | ... | 3.6 |
| | Normal | 38.5 | 41.5 | ... | 3.8 |
| | Slow | 43.0 | 45.5 | 48.0 | 5.5 |
| Simpson Logging Co., Shelton, Wash. | Fast | 28.5 | 26.5 | ... | 3.6 |
| | Normal | 34.0 | 29.5 | ... | 7.1 |
| | Slow | 44.5 | 40.0 | 39.0 | 8.0 |
| Wood Conversion Co., Cloquet, Minn. | Fast | 9.5 | 9.0 | ... | 2.7 |
| | Normal | 29.5 | 30.5 | ... | 1.7 |
| | Slow | 185.0 | 177.5 | 175.0 | 3.2 |

Accordingly, the instruments were equipped with a new trap, as shown in Fig. 1, and the tests were repeated on different samples of pulps.

## MATERIALS

The stocks used in this study consisted of a free, a normal, and a slow stock submitted by each of the following board manufacturers: Celotex Corp., Marrero, La.; Fir-Tex Insulating Board Co., St. Helens, Ore.; Flintkote Co., Meridian, Miss.; Johns-Manville Corp., Manville, N. J.; Minnesota and Ontario Paper Co., International Falls, Minn.; Simpson Logging Co., Shelton, Wash.; and Wood Conversion Co., Cloquet, Minn. A normal stock was submitted by Donnacona Paper Co., Donnacona, Que.

The pulps were sent to the Flintkote Research Laboratory, Whippany, N. J., from where a representative sample of each was packed in 2-gal. tin cans and sent to the Forest Products Laboratory, where they were stored at 39°F. until tested. The oven-dry content of the pulps ranged from 15 to 48%.

## EXPERIMENTAL

A weighed sample of the pulps (53.0 grams oven-dry equivalent) was soaked in tap water at room temperature for 1 hr. and then slushed for 30 to 60 sec. in the standard disintegrator described in TAPPI method T 205 m. From the disintegrator, the pulp was diluted with water at 80°F. to 5000 ml. in a 3-gal. container and thoroughly stirred. While stirring, four 1000-ml. samples were dipped into four graduates and the drainage rates were determined.

During the same period drainage rates on samples of the same pulps were determined at the Flintkote Research Laboratory with a similar SFMC tester. The first results obtained showed one tester to give erratic values with the slower pulps, which varied considerably from the values obtained with the other tester.

In attempts to determine the cause of these discrepancies, two normal pulps were stirred with the TAPPI disintegrator for different periods of time to see what effect this would have on the drainage time. Results of these tests, shown in Fig. 2, indicate that stirring does not affect some pulps appreciably, but may have a considerable effect on other pulps. This being the case, it is essential to standardize the length of time a pulp is stirred for the rate-of-drainage test.

Since any difference in the time of stirring could not have produced the wide variations in results obtained with one of the instruments, the effect of loss of vacuum in the water leg was determined.

Immediately after starting the drainage on a pulp with a drainage-time value of 60.5 sec., the union on the waterline between the water valve and the vacuum leg was loosened several turns, whereupon the drainage time increased to 286.5 sec. In another test, when the union was barely loosened, the drainage time increased slightly to 62.6 sec.

Similar but erratic results were obtained when small leaks were purposely formed under the gaskets holding the wire screen.

Since small leaks can affect the vacuum in the water

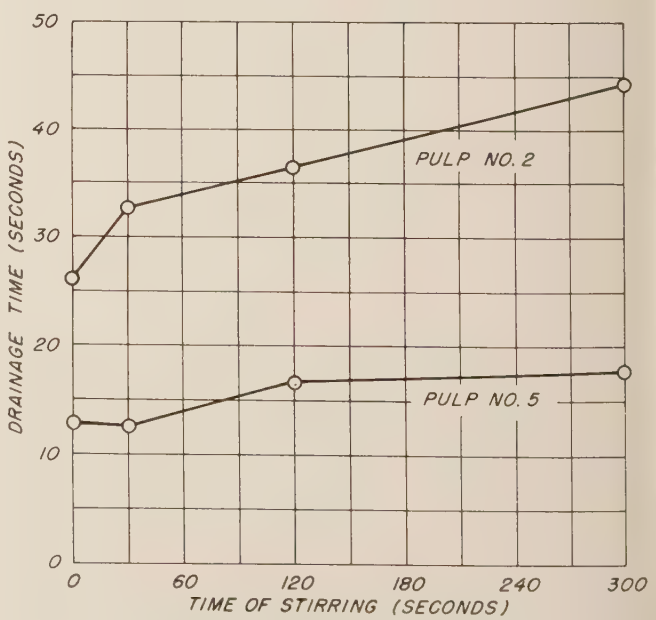


*Fig. 2. Effect of stirring on the drainage time of pulp*

leg so adversely, it is important that frequent checks be made to insure freedom from these leaks.

When new gaskets were installed in the tester that gave erratic results, the tests showed good agreement with the other tester as shown in Table I.

Table I also shows the reproducibility of results obtained by two operators with one tester. The tests were made with the slow pulps only, since they usually give the greatest spread in values.

## CONCLUSIONS

From the results of these tests, it appears that the two testers built in accordance with the specifications described in TAPPI method T 1002 sm-51 give comparable results for pulps given identical treatment. Stirring with a high-speed propeller-type stirrer alters some pulps sufficiently to change their drainage rates.

### *Recommendation*

More satisfactory conditions for the time and nature of stirring should be developed and specified in the standard method.

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# Studies of Rosin Sizing by Means of Autoradiography and Electron Microscopy

CHARLES W. HOCK

**The light microscope is an extremely useful tool for both research and control in the pulp and paper industry. There are, however, instances where the light microscope gives only meager and insufficient information. Visual studies of rosin sizing are a case in point. In order to obtain new information about this old problem, thereby giving a better answer to "How and in what form is rosin size distributed on the surfaces of fibers in a sheet of sized paper?", the electron microscope, with its higher resolving power, was utilized. Size precipitates, white water samples, and single fibers from sized sheets were examined. Direct electron microscopical examination of size precipitates and of white waters revealed that the particles of rosin size were more or less round, that they averaged about a thousand angstroms in diameter, and that they occurred either singly or in groups. Because single pulp fibers are too thick to be used directly in the electron instrument, the examination of their surfaces was accomplished by means of surface replication, wherein impressions of single fibers in thin films of plastic are used as specimens. The replication of objects as small as single wood pulp fibers is tedious. Fortunately, autoradiography (a technique of locating radioactive material by allowing a specimen to take its own picture) of single fibers from paper sized with radioactive rosin size showed that all the fibers were sized equally. This knowledge of fiber to fiber uniformity brought within practical limits the number of single fibers requiring replication. The electron micrographs revealed that the sized fibers had comparatively rough surfaces, owing to the fact that the fibers were irregularly covered with size particles similar to those seen previously in size precipitate and in white water. On the other hand, micrographs of fibers from unsized waterleaf sheets were smooth, except for the normal fibrillate surface pattern. These observations support the often expressed opinion—hitherto not adequately confirmed by direct visual observation—that rosin size is not present as smooth surface-covering films but rather in the form of discrete particles.**

In spite of a large amount of research carried out on rosin sizing of paper some aspects of the problem are not yet clearly understood. One of these pertains to the character and location of the size. Although not undisputed (*1*) it is pretty generally agreed that rosin size is attached to the fibers in the form of discrete particles and not as a continuous film (*8*). From studies with the light microscope Lee (*16*) showed, in drawings, the particulate distribution of size on fiber surfaces. By means of the same instrument, other investigators (*9*) have estimated the dimensions of particles of free rosin size and noted the influence of this property on the effectiveness of the size.

The light microscope is unquestionably a useful tool for both research and control in the pulp and paper industry, but because of its limited resolution, it sometimes gives only meager and insufficient information. Studies of rosin sizing are a case in point. On the other hand, the electron microscope, with its hundredfold increase in resolution, should be capable of yielding new information about this problem. A few investigators, notably Sears and Kregel (*17*), have used the electron microscope to examine cellulose and size, but since these early beginnings, improved electron microscopical techniques such as metal shadowing and replication have come into use, thereby warranting further studies, aimed at realizing the potential usefulness of electron microscopy in connection with problems of the paper industry.

In the present study the electron microscope was used to examine rosin size precipitates, sized cellulose fines from white water, and fibers from sheets of sized paper. Another new technique, viz., autoradiography, was used to obtain corroborative evidence which enhanced the value of the electron microscopical observations.

## EXPERIMENTAL PROCEDURE

### *Electron Microscopy*

For the most part, standard techniques of electron microscopy were used in preparing specimens for examination (*18*). Drops of aqueous suspensions of freshly prepared rosin size precipitate or of white water were placed on microscope slides which previously had been coated with a thin film of nitrocellu-

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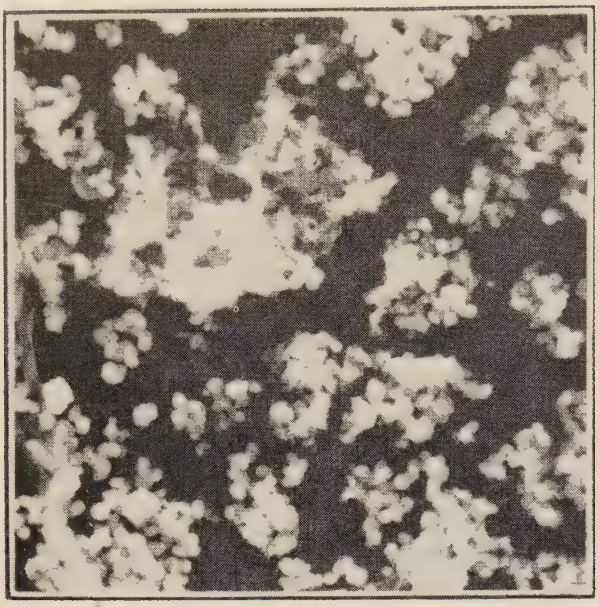

*Fig. 1. Electron micrograph of rosin size precipitate (×25,000)*

lose cast from amyl acetate. After the liquid evaporated the dry specimens were shadowed with chromium, at a low angle in order to increase contrast in the electron microscopical images. The shadowed specimens were floated from the glass slides onto water, captured on specimen screens, dried, and examined.

Inasmuch as specimens prepared according to the above procedure were air-dried, there was some uncertainty concerning whether or not changes in the extent of agglomeration of the size particles occurred during drying. In order to overcome this uncertainty specimens were also prepared by freeze-drying. After drops of the aqueous suspensions had been placed on the nitrocellulose-coated slides the droplets were frozen instantaneously by plunging the slides into liquid nitrogen. Subsequently, the slides were withdrawn and placed on a metal block cooled by dry ice. The cold block with the slides on it was placed in an evacuation unit where the moisture was removed from the

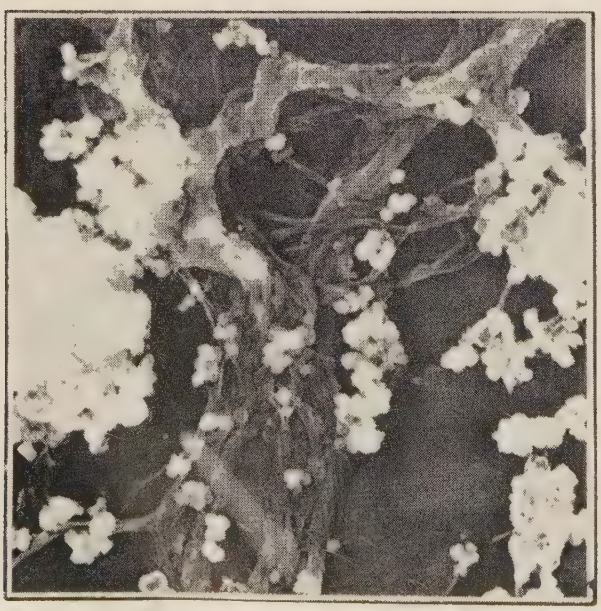

*Fig. 2. Electron micrograph showing cellulose and attached rosin size (×25,000)*

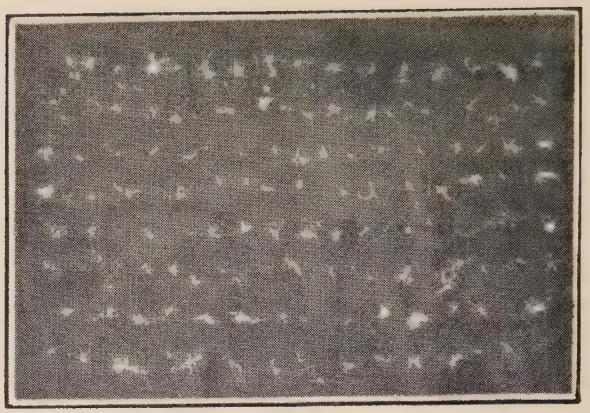

*Fig. 3. Autoradiograph of fibers from paper sized with radioactive rosin size (×1)*

still frozen specimens. Measurements of vapor pressure during evacuation revealed that during this operation the temperature did not rise above −40°C. After the moisture had been removed the specimens were shadowed with a beam of metallic vapor.

There were observable differences in the appearance of air-dried and frozen-dried specimens, but they were not of great magnitude. For critical work the specimens should probably be frozen before removal of the moisture, but for exploratory examinations air-dried specimens, which are less troublesome to prepare, yield worth-while information. The electron micrographs of size precipitate and of white water included here as illustrations are of frozen-dried specimens. In order to have pictures in which the metal shadows appear dark these micrographs were reversed photographically.

Unlike cellulose fines, which are obtained by sloughing off thin slivers of fiber, single pulp fibers cannot be examined directly in the microscope because they are many times too thick to permit the beam of electrons to pass through them. This difficulty can be circumvented by making surface replicas. Surface replicas of fibers drawn from sheets of paper were made as follows: A microscope slide was flooded with a 0.5% solution of nitrocellulose in amyl acetate. Before the film was completely dry individual pulp fibers were pressed against it. After the solvent had evaporated the fibers were stripped away, leaving their impressions in the plastic films, which were then shadowed with chromium at an angle of about 15°. Subse-

*Fig. 4. Photomicrograph of the fibers used in obtaining the autoradiograph shown in Fig. 3 (×1)*

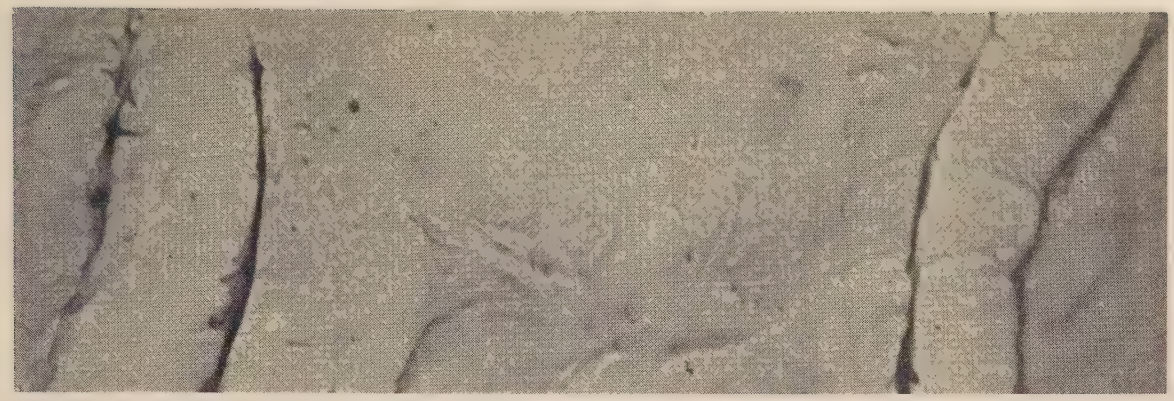

*Fig. 5. Electron micrograph of surface replica of fiber from unsized waterleaf (×25,000)*

quently, the films were removed from the slides and examined in the microscope. Electron micrographs of the fiber surface replicas were not reversed photographically.

*Autoradiography*

In autoradiography, a specimen containing a radioactive material is placed in contact with a suitable photographic film or plate which, after exposure and processing, reveals the location of the radioactive material within the specimen. The image obtained in this way is called an autoradiograph.

The radioelements which occur in nature commonly reach stability by successive emission of alpha particles. The synthetic isotopes, on the other hand, decay almost invariably by beta particle, positron, gamma-ray, or x-ray emission. All of these radiations produce a blackening on suitable photographic film. Fundamental principles, problems, techniques, and results of autoradiography are discussed in a book by Yagoda (*19*). Our present concern, however, is only with beta radiation. Some references pertaining specifically to beta particle autoradiography are listed in the bibliography (*2–7, 10–13, 15*). The beta particles are high-speed negatively charged electrons ejected from the nucleus of atoms in radioactive decay. Because the beta radiation is very weak, a highly sensitive photographic emulsion and intimate contact between the film and the specimen are essential for successful beta particle autoradiography.

A rosin size was prepared in such a way as to incorporate within it radioactive atoms which gave off beta radiation. Two per cent, by weight, of this labeled size was added to a slurry of bleached sulphite pulp, from which a handsheet was made. About half of the size was retained by the sheet, the rest was lost in the white water. Geiger counter measurements and calculations indicated that there were about 200 disintegrating atoms per sec. per sq. cm. of paper.

Single fibers and tufts of fibers were teased from the sheet of paper containing radioactive size and distributed on the sticky side of pieces of Scotch tape, which were then stuck on Kodak no-screen x-ray film. In order to ensure good contact between the film and the fibers a 5-lb. weight was placed on top of the film-fiber-tape sandwich. After exposures from one to

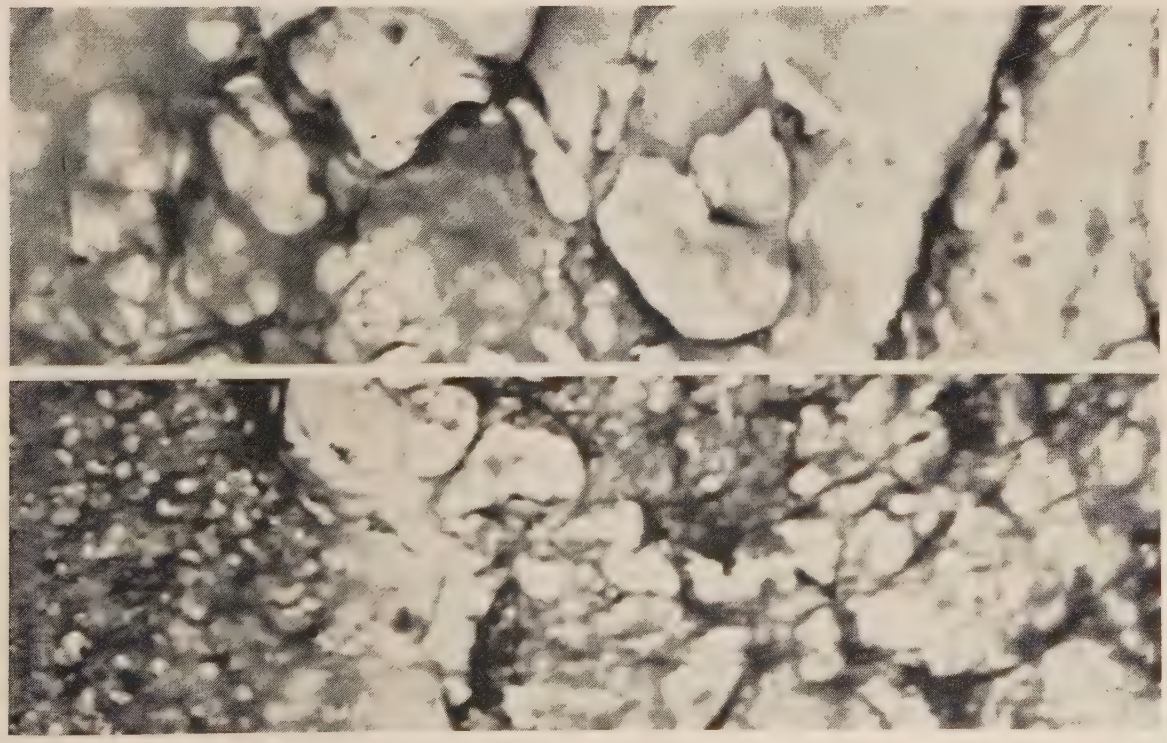

*Fig. 6. Electron micrographs of surface replicas of fibers from rosin-sized paper (×25,000)*

several weeks the films were developed in Kodak developer D-19.

## RESULTS AND DISCUSSION

The study of rosin sizing was begun by examining size precipitates. Figure 1 is an electron micrograph at a magnification of 25,000 times, showing particles of size precipitate obtained by adding alum to rosin size until a pH of 5 was reached. The resultant particles of aluminum resinate are more or less round and are either single or aggregated. Individual particles range in size from about 500 to 1500 A but they average about 1000 A.

White water was examined next in order to see how the size particles were situated on the cellulose fines. It is already well established from electron microscopical studies that these fines, like all native cellulose, are made up of fibrils a few hundred angstroms in diameter (*14*). Most of the particles of rosin size in the white water were in contact with the cellulose fibrils. As in the size precipitates, these size particles occurred either singly or in small or large groups (Fig. 2). The larger groups are, of course, big enough to be resolved by light microscopy, but that instrument would not resolve the small individual particles of which the clusters are composed. In the light microscope these groups would look like single large particles.

Electron microscopical examination of size precipitates and of samples of white water gave a clearer picture of the nature of the size particles, by revealing their shape, dimensional distribution, and tendency to agglomerate. Although this work was a necessary prerequisite to further studies, it did not lead directly to the main goal which was to find out how these particles are distributed on the surface of the fibers in a sheet of sized paper. In spite of the difficulties involved in replicating structures as small as single pulp fibers this seemed like the only way to get the desired information. But there is a deterrent to this approach to the problem. One might reasonably suspect that the individual fibers in a sheet of paper were sized to different extents. Since just 1 sq. in. of paper contains many hundreds of fibers it becomes impractical to examine enough of the fibers, by a method so tedious as replication, to get a significant composite picture of size distribution throughout the sheet. If there were a way of finding out how much fiber-to-fiber variation occurs in a sheet, the problem of sampling for replication might be simplified and the value of the results enhanced correspondingly.

Fortunately, there is a neat way of checking variations in the sizing level of individual fibers. Such a technique is autoradiography. Single fibers and small tufts of them were dissected from a sheet of paper sized with radioactive rosin size and held against film which was sensitive to the radiation given off by the disintegrating atoms. Figure 3 shows the result. The position of the fibers is clearly recorded. Figure 4 is a photograph of the very same fibers shown in the autoradiograph (Fig. 3). A comparison of these two pictures reveals that every fiber placed on the radiation-sensitive film recorded its position there. Moreover, all the fibers appear to have given off approximately equal amounts of radiation. The significant outcome of this test was, then, the proof that all the fibers from the paper were equally sized. Although the appearance of the autoradiographs indicated that all the fibers were evenly covered with size it does not follow that they are covered with continuous coatings of size. Owing to the limit of resolution of the film it is not certain, from autoradiography alone, whether the size forms a continuous coating or whether it is deposited as particles too small to be resolved by the photographic emulsion. But at least this experiment demonstrated that all the fibers in the sheet of paper were sized equally. With this information it became possible to replicate only a comparatively small number of fibers for electron microscopical study, knowing that those fibers were representative of the condition in the entire sheet.

Before looking at the replicas of sized fibers it is necessary to know, also, what fibers from unsized waterleaf sheets look like. Figure 5 shows such a fiber. The fiber surface is comparatively smooth, except for a faint fibrillate pattern of the sort one would expect to find in unsized pulp fibers (*14*). On the other hand, fibers from sized paper are distinctly different. Their surfaces are rough, owing to the fact that they are more or less covered with spheroidal size particles (Fig. 6) like those observed previously in size precipitate and in white water. Again, the diameter of the size particles averages about 1000 A, the larger agglomerates being aggregates of the smaller ones. A comparison of the upper and lower illustrations in Fig. 6 gives an indication of the minor variations in the sizing level of individual fibers.

The results of this study confirm the idea that rosin size is attached to fibers in the form of discrete particles and that these particles, at least under some circumstances, are distributed with remarkable uniformity among all the fibers in a sized sheet. The success achieved in this preliminary study of rosin sizing also points up the potential usefulness of electron microscopy for exploring other aspects of the sizing problem.

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# Sulphite Spent Liquor V

## *Equilibrium Distribution of Ammonium and Other Ions Between Cation Exchange Resins and Aqueous Solutions*

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**The equilibrium distribution of ammonium and hydrogen ions between cation exchange resins and sulphite spent liquor or sulphurous acid solutions has been determined as a basis for consideration of processes for recovery of ammonia from sulphite spent liquor by adsorption on a cation exchange resin followed by stripping with a solution of sulphurous or some other acid under various conditions. The results are correlated satisfactorily by the treatment of Bauman and Eichhorn using an ammonium-hydrogen ion selectivity coefficient of about 1.2 when the ion fraction of conversion to ammonium resin is not above about 0.7. Some data for calcium-hydrogen ion exchange are also reported. In general, it appears probable that the equilibrium distributions for the sodium-hydrogen, and for certain other ion exchange processes can be estimated with moderately good accuracy simply by carrying out calculations using the selectivity coefficients given by Bauman and Eichhorn and others.**

AMMONIUM ions can be removed from ammonium sulphite spent liquor by adsorption onto an appropriate cation exchange resin. The adsorbed ions can then in part be stripped from the resin by use of a sulphurous acid solution whereby the resin is regenerated, and part of the ammonia is recovered as a bisulphite solution possibly suitable for reuse. Preliminary experimental investigation of the process in this Laboratory has shown that about 60% of the ammonium ion present can be recovered under certain conditions of operation of the cycle (*1*).

The extent of ammonia recovery attainable by this type of treatment is determined by the way ammonium ions under equilibrium conditions are distributed between ammonium sulphite spent liquor and resin, and between the resin and sulphurous acid solution. Thus, the present work has been carried out to evaluate the distribution of ammonium ion between sulphite spent liquor or sulphurous acid solutions and several cation exchange resins. Some information has also been obtained for the calcium-hydrogen ion exchange process.

### EXPERIMENTAL

#### *Solutions*

Three sulphite spent liquors were used in the experiments. Sample A was a paper pulp ammonium sulphite spent liquor (kindly provided by W. W. Moyer of the Crown Zellerbach Corp.), containing total solids at about 14% concentration and of a composition as previously reported (*1*). This liquor was concentrated by evaporation at atmospheric pressure to about 46% solids concentration, partly de-ashed by use of a cation exchange resin, and then used for equilibriums studied with the Dowex 50-8X resin.

Sample B was a steam stripped, paper pulp calcium sulphite spent liquor (kindly provided by E. O. Ericsson of the Puget Sound Pulp & Timber Co.) of a composition about the same as previously reported (*2*). This liquor was concentrated by vacuum evaporation to 29% solids concentration, completely de-ashed by treatment with a cation exchange resin, and then neutralized with ammonium hydroxide to pH 4.2 to provide an ammonium sulphite spent liquor for equilibrium studies with the Duolite C-60 resin.

Sample C was an unstripped, paper pulp calcium sulphite spent liquor, also kindly provided by E. O. Ericsson, containing 94 grams total solids and 6.12 grams CaO per liter. An aliquot of this liquor was de-ashed in part, using special care to avoid dilution, and was then designated as sample CD.

The sulphurous acid solutions used were prepared by bubbling sulphur dioxide into distilled water.

#### *Resins*

Experiments were carried out using the following types of cation exchange resins: (1) polystyrene nuclear sulphonate resin Dowex 50 containing 8% divinyl benzene (Dow Chemical Co., Midland, Mich.); (2) polystyrene nuclear sulphonate resin Dowex 50 containing 1% divinyl benzene and at 50–100 mesh (Dow Chemical Co.); (3) polystyrene methylene sulphonate resin Amberlite IR 100 (Rohm & Haas Co., Philadelphia, Pa.); (4) phosphonic cation exchange resin, Duolite C-60 (Chemical Process Co., Redwood City, Calif.); (5) a lignin sulphonate-formaldehyde cation exchange resin prepared in this Laboratory (3).

For the ammonia equilibrium studies, the resins were used as ammonium resins for experiments 1A to 9B inclusive and as hydrogen resins for all other experiments. The ammonium resins were prepared from the hydrogen resins by treating exhaustively with ammonium hydroxide, washing until nearly neutral, and draining off the excess water by suction filtration.

For the calcium equilibrium studies, Amberlite IR 100 was used as the hydrogen resin. A pretreatment of the hydrogen resin with calcium sulphite spent liquor was made before using the resin in experiments 17 to 20 inclusive.

#### *Procedure for Ammonia Distribution Experiments*

The desired weight of moist resin was placed in a 125-ml. glass stoppered Erlenmeyer flask, and then 50 ml. or some other appropriate volume of sulphurous

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acid solution or sulphite spent liquor was pipetted in. The flask was stoppered, placed in a 25°C. water bath, and equilibrated for 2 to 4 hr. with swirling of the contents at 15-min. intervals.

After equilibration, an appropriate aliquot of the liquid phase was removed by pressure pipetting and diluted to a known volume and analyzed to determine sulphur dioxide and ammonia concentration for the sulphurous acid-resin experiments. Analysis for ammonia concentration only was carried out in the sulphite spent liquor-resin experiments.

Total sulphur dioxide was determined by TAPPI Standard T 604 m-45. Ammonia was determined by adding sodium hydroxide to make the solution alkaline, distilling the evolved ammonia into an excess of standardized solution of acid, and then back titrating the remaining acid with a standardized solution of sodium hydroxide.

Immediately after withdrawing a liquid sample from an equilibration flask, the resin was removed by filtration on a sintered glass Buchner funnel, and then quickly washed with three portions of distilled water to try to remove surface liquid only. The Dowex 50-8X resins from the sulphite spent liquor experiments were analyzed for ammonia content by weighing a quantity of the resin of known moisture content into a distillation flask, adding 200 ml. of 1 *N* NaOH solution and then distilling off the evolved ammonia as described above. The total capacity of this resin was calculated from the known capacity per gram of moisture-free resin and moisture content determined by drying a sample of the resin at 105°C. for 24 hr.

In all other experiments the resin was placed in a small glass column and stripped of all ammonia by elution with 1 *N* HCl, and then washed with distilled water until free of acid. Ammonia was determined in this effluent plus washings solution by the procedure outlined above. The total capacity of the resultant hydrogen resin was measured by placing the entire resin in a flask, adding an excess of sodium chloride, and titrating to the phenolphthalein end point with standardized sodium hydroxide solution.

This method for determination of resin capacity was satisfactory for use with all of the resins studied except the phosphonic resin Duolite C-60. This resin was converted to the sodium form by column treatment with sodium hydroxide, and washed until free of base. A known three-fold excess of standard hydrochloric acid was then passed through the column of resin, and an aliquot of the effluent was back titrated with standardized basic solution. The loss in equivalents of acid by passage of the acid through the resin column is a measure of the total available sites on the resin.

### *Procedure for Calcium Distribution Experiments*

For calcium distribution with respect to sulphurous acid, the moist Amberlite IR 100 hydrogen resin was placed in a 250-ml. glass stoppered bottle and the bottle was filled with liquor C and allowed to stand several hours. The bottle containing the resin was then drained and filled with water and this was repeated. To the resultant pretreated resin was added an appropriate amount of calcium carbonate, and then sufficient sulphurous acid solution to fill the bottle. The bottle was stoppered, placed in a 30±0.5°C. water bath on a "ferris wheel" rotating at 4 r.p.m. and allowed to equilibrate for 48 hr.

For the distribution of calcium into sulphite spent liquor, the moist Amberlite IR 100 hydrogen resin was placed in a 250-ml. glass stoppered bottle together with the appropriate volumes of sulphite spent liquors C and CD and the system was permitted to stand at 30°C. for 96 hr.

After equilibration, the liquid phase was siphoned off and the resin was filtered onto a sintered glass filter and washed with 250 ml. of water. Analysis for calcium in the solution and on the resin was carried out by a procedure reported earlier (*4*). Resin samples were ashed for the calcium determination. The total resin capacity was measured by saturating a resin with calcium ions and then determining the calcium content of the resin as usual.

## DISCUSSION

The ammonium-hydrogen ion exchange process can be written as follows:

$$ {}_{S}NH_4^+ + {}_{R}H^+ \leftrightharpoons {}_{S}H^+ + {}_{R}NH_4^+ \qquad (1) $$

For absorption of ammonium ions from sulphite spent liquor onto a cation exchange resin in exchange for hydrogen ions, it is desired that the process should proceed from left to right, and for stripping of ammonium ions from the resin into an acidic solution, the process must be made to go in the opposite direction so as to give the products shown on the left side of equation (1). The net result of these processes is that ammonium and hydrogen cations are exchanged between an aqueous solution phase, where they coexist with lignin sulphonate, bisulphite, or other mobile anions, and an ion exchange resin phase where anionic groups are fixed to the resin structure.

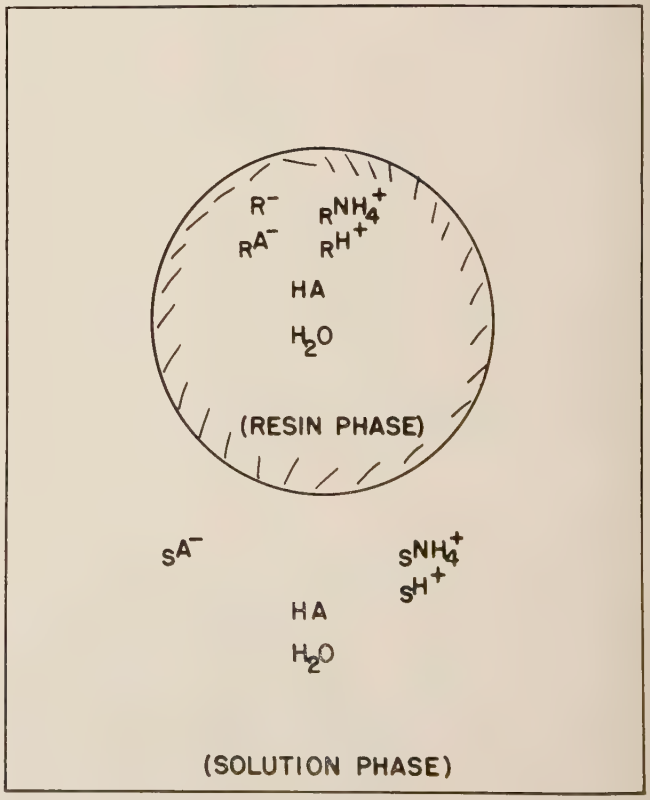


*Fig. 1. Schematic representation of ion exchange equilibrium*

**Table I. Equilibrium Distribution of Ammonium Ions Between Sulphite Spent Liquor and Cation Exchange Resins**

| | Added components | | | | Equilibrium composition $NH_3$ Balance | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Conc. SSL[c] | | | | | Resin | | | | |
| Expt. | Resin type[a] | Wt.,[b] g. | Vol., ml. | $NH_3$, g./l. | $NH_4OH$ Soln.[e] ml. | Soln., $NH_3$, g./l. | $NH_3$, m.eq. | Sites,[f] m.eq. | $NH_3$ input, m.eq. | $NH_3$ found, m.eq. | Ion fractions $X_S$[g] | $X_R$ |
| 157-3 | D-50-8 | 12 | 100 | 7.87 | 0 | 4.71 | 19.8 | 59.0 | 46.2 | 47.5 | 0.24 | 0.31 |
| 157-1 | D-50-8 | 2 | 100 | 7.87 | 0 | 7.11 | 4.3 | 9.84 | 46.2 | 46.1 | 0.36 | 0.45 |
| 157-4 | D-50-8 | 5 | 100 | 7.87 | 1 | 7.92 | 11.6 | 24.6 | 61.2 | 58.6 | 0.41 | 0.47 |
| 157-6 | D-50-8 | 5 | 100 | 7.87 | 4 | 13.0 | 16.7 | 24.6 | 106.2 | 96.1 | 0.69 | 0.68 |
| 157-2 | D-50-8 | 5 | 100 | 7.87 | 0 | 6.16 | 9.85 | 24.6 | 46.2 | 46.0 | 0.32 | 0.40 |
| 157-5 | D-50-8 | 5 | 100 | 7.87 | 2 | 8.99 | 12.9 | 24.6 | 76.2 | 66.7 | 0.47 | 0.52 |
| 157-7 | D-50-8 | 5 | 100 | 7.87 | 6 | 15.77 | 19.4 | 24.6 | 136 | 117.4 | 0.85 | 0.79 |
| 7A | Duo.-C60 | 5 | 50 | 11.5 | 0 | 10.5 | 2.60 | 13.4 | 33.8 | 33.5 | 0.91 | 0.22 |
| 7B | Duo.-C60 | 20 | 50 | 11.5 | 0 | 9.03 | 5.96 | 53.0 | 33.8 | 32.5 | 0.78 | 0.14 |

[a] D-50-8 designates the Dow Chemical Co. cation exchange resin Dowex 50-8% DVB, and Duo-C60 the Chemical Process Co. cation exchange resin, Doulite C-60.
[b] D-50-8 weights are on oven-dry basis.
[c] Refer to "Experimental" section for description of sulphite spent liquor used.
[d] Alkali ammonia.
[e] 15 *N* ammonium hydroxide solution was added.
[f] Accepting Bauman's figure of 202 for equivalent weight of dry resins.
[g] $X_S'$ and $X_S$ were calculated assuming 1 equivalent of anionic sites for each 400 grams of total solids.

A synthetic ion exchange resin such as Dowex 50 consists of a three-dimensional network of sulphonated polystyrene molecules cross linked with divinyl benzene (*5*). This network may be sufficiently loose to permit easy passage in and out of the resin of cations such as hydrogen and ammonium. However, the sulphonic acid groupings are attached to the resin skeleton, and thus maintain within this skeleton an electrolyte concentration which is determined by the frequency of occurrence and the degree of dissociation of the ionic groups present. In Dowex 50 a high electrolyte concentration exists because many sulphonate groups are present per unit volume and because cations are nearly completely dissociated from these groups.

This region of high electrolyte concentration within a resin particle has been considered to be a phase which is different from the outside solution phase. On this basis Bauman and Eichhorn (*5*) have treated ion exchange as a heterogeneous process governed by the Donnan theory of membrane equilibria (*6*) between the outside solution phase and the resin gel phase and their treatment will now be summarized and applied to correlate the equilibrium data obtained experimentally.

At equilibrium as represented in Fig. 1, the chemical potentials of the substances present on both sides of the membrane must be equal, and since the chemical potential of a strong electrolyte may be taken as the sum of the potentials of its ions (*7*), then equations (2) and (3) are written:

$$({}_R a_{NH_4})({}_{R\cdot}\,{}_A) = ({}_S a_{NH_4})({}_S a_A) \quad (2)$$

$$({}_R a_H)({}_R a_A) = ({}_S a_H)({}_S a_A) \quad (3)$$

These equations are rearranged and terms representing concentrations and activity coefficients are substituted for the activities to obtain:

$$\frac{{}_S a_A}{{}_R a_A} = \frac{{}_R a_{NH_4}}{{}_S a_{NH_4}} = \frac{{}_R a_H}{{}_S a_H} = \frac{({}_R C_{NH_4})({}_R \gamma_{NH_4})}{({}_S C_{NH_4})({}_S \gamma_{NH_4})} = \frac{({}_R C_H)({}_R \gamma_H)}{({}_S C_H)({}_S \gamma_H)} \quad (4)$$

If the aqueous solution outside the resin particle is dilute then ${}_S\gamma_H/{}_S\gamma_{NH_4} \cong 1$ (*8*), so there results:

$$\frac{({}_R C_{NH_4})({}_S C_H)({}_S \gamma_H)}{({}_S C_{NH_4})({}_R C_H)({}_S \gamma_{NH_4})} = \frac{({}_R C_{NH_4})({}_S C_H)}{({}_S C_{NH_4})({}_R C_H)} = \frac{({}_R \gamma_H)}{({}_R \gamma_{NH_4})} = K \quad (5)$$

Alternatively, equation (5) can be expressed as:

$$\left(\frac{X_R}{1 - X_R}\right) = K\left(\frac{X_S}{1 - X_S}\right) \quad (6)$$

where $X_R$ or $X_S$ is the fraction of the total anionic sites

**Table II. Equilibrium Distributions of Ammonium Ions Between Sulphurous Acid Solutions and Cation Exchange Resins**

| | Added components | | | | Equil. composition | | | | Material balances | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Resin | | $SO_2$ soln. | | Soln. | | Resin | | $SO_2$ | | $NH_3$ | | Ion fractions | | |
| Expt. | Type[a] | Wt.,[b] g. | Vol., ml. | Conc., g./l. | $SO_2$, g./l. | $NH_3$, g./l. | $NH_3$, m.eq. | Sites, m.eq. | Added, mmols. | Found, mmols. | Soln. found, m.eq. | Resin calc.,[c] m.eq. | $X_S'$ | $X_S$ | $X_R$ |
| 2A | D-50-8 | 5.0 | 50 | 66.0 | 55.2 | 1.66 | 7.80 | 12.7 | 51.5 | 45.2 | 4.88 | 7.82 | 0.11 | 0.63 | 0.62 |
| 2B | D-50-8 | 5.0 | 50 | 66.0 | 53.3 | 1.65 | 7.85 | 12.9 | 51.5 | 43.8 | 4.85 | 8.05 | 0.12 | 0.63 | 0.63 |
| 2C | D-50-8 | 20.0 | 50 | 66.0 | 46.6 | 3.14 | 40.8 | 51.9 | 51.5 | 43.6 | 9.24 | 42.66 | 0.25 | 0.86 | 0.82 |
| 2D | D-50-8 | 20.0 | 50 | 66.0 | 46.2 | 3.14 | 39.1 | 51.3 | 51.5 | 43.4 | 9.24 | 43.06 | 0.26 | 0.86 | 0.84 |
| 9A(1°C.) | D-50-8 | 5.0 | 50 | 64.0 | 54.4 | 1.74 | 7.70 | 13.1 | 50.0 | 44.6 | 5.11 | 7.99 | 0.12 | 0.55 | 0.61 |
| 9B(1°C.) | D-50-8 | 20.0 | 50 | 64.0 | 47.3 | 3.41 | 39.2 | 52.4 | 50.0 | 44.4 | 10.10 | 42.3 | 0.27 | 0.93 | 0.81 |
| 3H | D-50-8 | 1.0 | 165 | 12.8 | 11.9 | 0.187 | 0.839 | 2.63 | 33.0 | 30.8 | 1.81 | 0.82 | 0.059 | 0.24 | 0.31 |
| 3I | D-50-8 | 1.0 | 165 | 12.8 | 11.8 | 0.190 | 0.853 | 2.68 | 33.0 | 30.6 | 1.85 | 0.83 | 0.061 | 0.24 | 0.31 |
| 3F | D-50-8 | 1.0 | 75 | 12.8 | 11.9 | 0.340 | 1.21 | 2.70 | 15.0 | 14.1 | 1.50 | 1.20 | 0.11 | 0.38 | 0.45 |
| 3G | D-50-8 | 1.0 | 75 | 12.8 | 11.9 | 0.330 | 1.14 | 2.59 | 15.0 | 14.1 | 1.46 | 1.13 | 0.10 | 0.37 | 0.44 |
| 3A | D-50-8 | 5.0 | 50 | 12.8 | 11.0 | 1.04 | 9.86 | 13.05 | 10.0 | 9.04 | 3.05 | 10.00 | 0.36 | 0.80 | 0.77 |
| 3B | D-50-8 | 5.0 | 50 | 12.8 | 11.0 | 1.03 | 9.58 | 12.72 | 10.0 | 9.04 | 3.03 | 9.69 | 0.35 | 0.79 | 0.76 |
| 3E | D-50-8 | 5.0 | 50 | 12.8 | 10.6 | 1.04 | 10.2 | 13.17 | 10.0 | 8.67 | 3.06 | 10.11 | 0.37 | 0.80 | 0.77 |
| 3C | D-50-8 | 20.0 | 50 | 12.8 | 10.2 | 1.68 | 46.1 | 51.75 | 10.0 | 9.58 | 4.95 | 46.80 | 0.62 | 0.94 | 0.91 |
| 3D | D-50-8 | 20.0 | 50 | 12.8 | 9.94 | 1.67 | 45.5 | 50.83 | 10.0 | 9.32 | 4.91 | 45.92 | 0.63 | 0.94 | 0.90 |
| 1A | D-50-1 | 5.0 | 50 | 67.0 | 53.4 | 0.726 | 0.862 | 2.96 | 52.4 | 43.9 | 2.13 | 0.83 | 0.051 | 0.36 | 0.28 |
| 1B | D-50-1 | 5.0 | 50 | 67.0 | 51.9 | 0.713 | 0.951 | 2.77 | 52.4 | 42.6 | 2.09 | 0.68 | 0.052 | 0.30 | 0.25 |
| 1C | D-50-1 | 20.0 | 50 | 67.0 | 42.9 | 1.55 | 6.03 | 12.16 | 52.4 | 40.2 | 4.56 | 7.60 | 0.14 | 0.65 | 0.63 |
| 1D | D-50-1 | 20.0 | 50 | 67.0 | 43.2 | 1.56 | 5.68 | 12.10 | 52.4 | 40.5 | 4.59 | 7.51 | 0.14 | 0.65 | 0.62 |
| 1E[d] | D-50-1 | 20.0 | 50 | 66.5 | 45.7 | 7.23 | 10.33 | 12.32 | 52.0 | 44.4 | 22.1 | | 0.60 | 0.98 | 0.84 |
| 8A | D-50-1 | 5.0 | 50 | 12.5 | 10.3 | 0.535 | 1.33 | 2.94 | 9.76 | 8.45 | 1.57 | 1.37 | 0.20 | 0.57 | 0.47 |
| 8B | D-50-1 | 20.0 | 50 | 12.5 | 8.89 | 0.935 | 8.13 | 11.70 | 9.76 | 8.34 | 2.75 | 8.95 | 0.40 | 0.81 | 0.77 |
| A | A-100 | 5.0 | 50 | 65.0 | 47.0 | 0.975 | 2.89 | 5.41 | 50.8 | 38.6 | 2.87 | 2.54 | 0.078 | 0.47 | 0.47 |
| 5B | A-100 | 20.0 | 50 | 65.0 | 32.9 | 1.947 | 15.5 | 22.6 | 50.8 | 30.9 | 5.73 | 16.9 | 0.22 | 0.76 | 0.75 |
| 5A | D-C-60 | 5.0 | 50 | 65.0 | 50.4 | 2.73 | 0.447 | 13.4 | 50.8 | 41.4 | 8.03 | 5.37 | 0.20 | 0.81 | 0.40 |
| 4B | D-C-60 | 20.0 | 50 | 65.0 | 41.0 | 8.11 | 6.49 | 53.0 | 50.8 | 38.5 | 23.9 | 29.1 | 0.74 | 0.99 | 0.55 |
| 4A | LS-F | 5.0 | 50 | 65.0 | 49.4 | 0.70 | 0.613 | 2.13 | 50.8 | 40.5 | 2.06 | 0.07 | 0.053 | 0.36 | 0.29 |

[a] Resins listed here as D-50-8, D-50-1, A-100, D-C-60, and LS-F refer to Dowex 50-8X, Dowex 50-1X, Amberlite IR 100, Duolite C-60, and lignin sulphonate-formaldehyde resin respectively.
[b] Weights given here were taken on the resins after drying only to the extent of drawing air through the resins for 5 min. on a suction filter.
[c] Obtained by assuming resin sites to be saturated with ammonia at start and then subtracting the m.eq. of ammonia in solution from the total sites as found by analysis.
[d] Two milliliters of 15 *N* $NH_4OH$ were added.

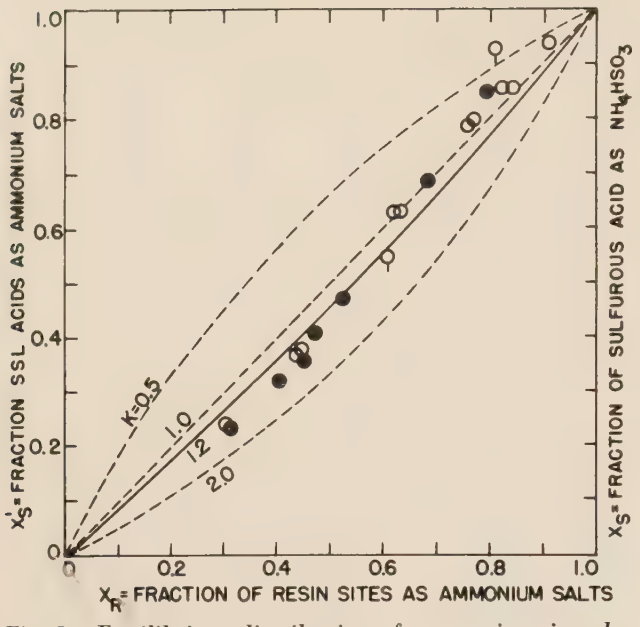


*Fig. 2. Equilibrium distribution of ammonium ions between solutions and Dowex 50–8% DVB. ○, sulphurous acid, 25°C; ♀, sulphurous acid, 1°C.; ●, sulphite spent liquor*

in the resin or solution $C_R$ or $C_S$, which are occupied by ammonium ions, respectively;

$$X_R = \frac{({}_RC_{NH_4})}{({}_RC_{NH_4} + {}_RC_H)} = \left(\frac{{}_RC_{NH_4}}{C_R}\right) \quad (7)$$

$$X_S = \frac{({}_SC_{NH_4})}{({}_SC_{NH_4} + {}_SC_H)} = \left(\frac{{}_SC_{NH_4}}{C_S}\right) \quad (8)$$

The value of the ion exchange selectivity coefficient $K$ for the distribution of ammonium and hydrogen ions has been found by Bauman and Eichhorn (*5*) to be about 1.20 when the solution phase is dilute. This value has been used in equation (6) to establish the relation between $X_S$ and $X_R$ shown as a full line in Fig. 2. The dotted lines given in Figs. 2 and 3 are also obtained from equation (6) for other values of $K$ as designated.

To apply these relations to the correlation of the experimental results, use has been made of the known or determined concentration of ammonia and of total lignin sulphonate or sulphurous acid in the solutions as shown in Tables I and II. The ratios of the total moles of ammonia to total moles of sulphonate or "sulphur dioxide" in the solutions have been calculated and are represented in the Tables I and II by the symbol $X'_S$.

However to consider the data with respect to the selectivity coefficient, the following ionization reactions and equilibria must be taken into account:

$$H_2SO_3 \leftrightharpoons H^+ + HSO_3^- \qquad K_{H_2SO_3} \cong \frac{(C_H)(C_{HSO_3})}{(C_{H_2SO_3})} \quad (9)$$

$$LSO_3H \leftrightharpoons H^+ + LSO_3^- \qquad K_{LSH} \cong \frac{(C_H)(C_{LS})}{(C_{LSH})} \quad (10)$$

$$LSO_3NH_4 \leftrightharpoons NH_4^+ + LSO_3^- \qquad K_{LSNH_4} \cong \frac{(C_{NH_4})(C_{LS})}{(C_{LSNH_4})} \quad (11)$$

The experiments of Johnstone and Leppla (*9*) have been used (*10*) to establish the following equation for the ionization constant for the first hydrogen from sulphurous acid:

$$\log_{10} K_{H_2SO_3} = \frac{843.33}{273.1 + t} - 4.71507 \quad (12)$$

when temperature is $t$°C. Ammonium bisulphite is assumed to be completely dissociated. Ernsberger and France (*11*) give data for the extent of dissociation of different salts of lignin sulphonates.

The computation of the ion fractions of conversion

## NOTATION

| | |
|---|---|
| $a$ | = molar activity of a component |
| $C$ | = molar concentration of a component |
| $C_R$ | = total molar exchange capacity of the resin |
| $C_S$ | = total molar concentration of the solution |
| $K$ | = dissociation constant (with subscript) |
| $K$ | = equilibrium constant or selectivity coefficient (without subscript) |
| $X_R$ | = mole fraction of sites occupied by ammonium or calcium ions to total anionic sites on the resin |
| $X_S$ | = mole fraction of ammonium or calcium ions to total anionic sites in solution |
| $X_S'$ | = mole fraction of total ammonia or calcium to total "sulphur dioxide" or total lignin sulphonate in the solution |
| $\gamma$ | = molar activity coefficient of a component |

## SUBSCRIPTS*

| | |
|---|---|
| A | = anion |
| H | = hydrogen ions |
| $HSO_3$ | = bisulphite ions |
| $H_2SO_3$ | = undissociated sulphurous acid |
| LS | = lignin sulphonate ions |
| $LSCa_{1/2}$ | = undissociated calcium lignin sulphonate |
| LSH | = undissociated hydrogen lignin sulphonate |
| $LSNH_4$ | = undissociated ammonium lignin sulphonate |
| R | = resin phase |
| S | = solution phase |
| TCa | = total molar concentration of calcium in solution |
| TLS | = total molar concentration of lignin sulphonates in solution |
| $TNH_4$ | = total molar concentration of ammonia in solution |
| $TSO_2$ | = total molar concentration of sulphurous acid in solution |

* Where two subscripts seem necessary, one has been placed before and the other after the symbol modified.

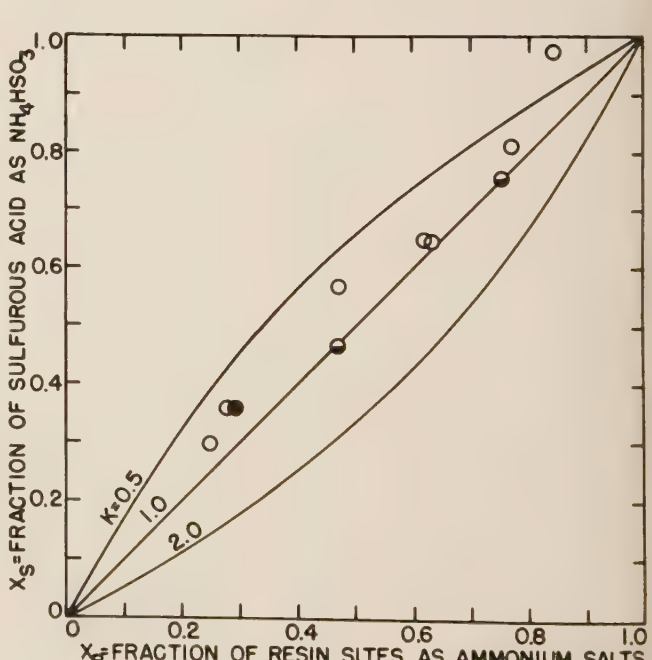


*Fig. 3. Equilibrium distribution of ammonium ions between sulphurous acid solutions and cation exchange resins at 25°C. ○, Dowex-50-1% DVB; ◒, Amberlite IR 100; ●, laboratory resin prepared from lignin sulphonates and formaldehyde*

**Table III. Equilibrium Distribution[a] of Calcium Ions Between Aqueous Solutions and Amberlite IR 100**

| Expt. | Added components[b] SSL-C, ml. | SSL-CD, ml. | $CaCO_3$, g. | $H_2SO_3$[c] ml. | Equilibrium compositions Soln., mmols. Ca/l. | Resin, mmols. Ca | Calcium balance Added, mmols. | Found, mmols. | Calcium ion fractions $X_S'$[e] | $X_S$[e] | $X_R$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 5 | 235 | 0 | 0 | 3.09 | 0.36 | .. | 2.2 | 0.013 | .. | 0.164 |
| 2 | 15 | 225 | 0 | 0 | 9.22 | 0.48 | .. | 5.4 | 0.039 | .. | 0.218 |
| 3 | 60 | 180 | 0 | 0 | 30.6 | 0.66 | .. | 16.0 | 0.130 | .. | 0.299 |
| 4 | 240 | 0 | 5.25 | 0 | 113.8 | 0.99 | 52.5 | 56.5 | 0.485 | .. | 0.448 |
| 17[d] | 0 | 0 | 0 | 235 | 1.62 | 0.40 | 0.79 | 0.8 | 0.002 | 0.016 | 0.182 |
| 18[d] | 0 | 0 | 0.21 | 235 | 8.64 | 0.56 | 2.8 | 2.6 | 0.010 | 0.081 | 0.253 |
| 19[d] | 0 | 0 | 1.23 | 235 | 46.4 | 0.95 | 13.1 | 12.1 | 0.051 | 0.306 | 0.430 |
| 20[d] | 0 | 0 | 4.11 | 235 | 163 | 1.04 | 41.9 | 40.2 | 0.178 | 0.472 | 0.473 |

[a] Experiments were conducted at 30 ± 0.5°C.
[b] Resin used was equivalent to 1 gram oven dry.
[c] Sulphurous acid solution contained 58.5 g. $SO_2$ per liter.
[d] Resin used here was pretreated with sulphite spent liquor C and then thoroughly washed with water. Calcium introduced by the resin is included in the balance calculations.
[e] $X_S'$ and $X_S$ were calculated assuming 1 equivalent of anionic sites per 400 grams of total solids.

$X_S$ is carried out by recognizing that the positive and negative charges in solution must be equal:

$$C_H + C_{NH_4} = C_{HSO_3} + C_{OH} \quad (13)$$
$$C_H + C_{NH_4} = C_{LS} + C_{OH} \quad (14)$$

and after neglecting the hydroxyl ion concentration, that the sum of the ionized and the nonionized lignin sulphonate or sulphurous acid particles must equal the total present,

$$C_{TSO_2} = C_{HSO_3} + C_{H_2SO_3} \quad (15)$$
$$C_{TLS} = C_{LS} + C_{LSH} + C_{LSNH_4} \quad (16)$$

Substituting these relations into equations (9), (10), and (11) gives for the sulphurous acid solutions;

$$C_{HSO_3}^2 + C_{HSO_3}(K_{H_2SO_3} - C_{NH_4}) - (K_{H_2SO_3}C_{TSO_2}) = 0 \quad (17)$$

and for the ammonium lignin sulphonate solutions;

$$C_{LS}^3 + C_{LS}^2(K_{LSH} + K_{LSNH_4}) + C_{LS}(K_{LSH}K_{LSNH_4} - K_{LSH}C_{TLS} + K_{LSH}C_{TNH_4} - K_{LSNH_4}C_{TNH_4}) - (K_{LSH}K_{LSNH_4}C_{TLS}) = 0 \quad (18)$$

These two equations can be used with appropriate values of $K_{H_2SO_3}$, $K_{LSH}$, and $K_{LSNH_4}$ and with known or experimentally determined concentrations of total ammonia or "sulphur dioxide" or lignin sulphonates, to solve for the equilibrium concentration of $HSO_3$ or $LS$

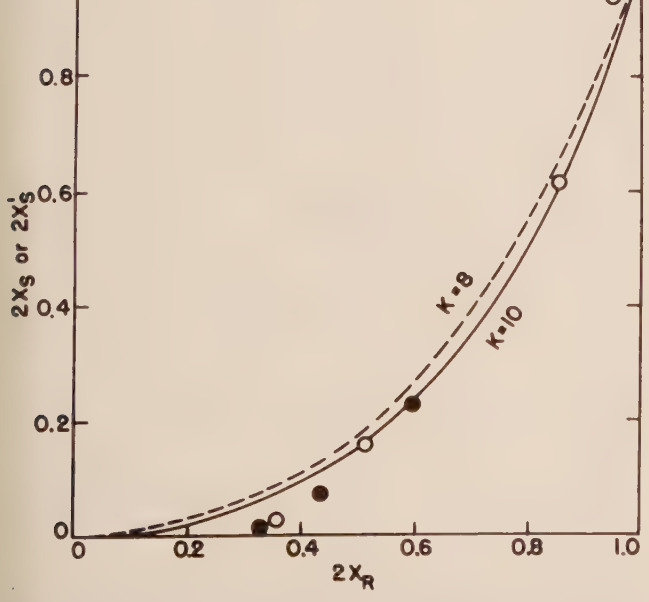


*Fig. 4. Equilibrium distribution of calcium ions between solutions and Amberlite IR 100. ○, sulphurous acid; ●, sulphite spent liquor; —, calculated for K = 10; - - -, calculated for K = 8*

Dissociation constants for lignin sulphonates calculated from data of Ernsberger and France (*11*) are quite low and also change with concentration. Lignin sulphonates are probably almost completely dissociated. Since the dissociation constants for lignin sulphonates are questionable, only the cation fraction of ammonium ions in the sulphurous acid solutions, $X_S$, were calculated with results given in Table II.

Values of $X_S'$ found for lignin sulphonate and of $X_S$ found for sulphurous acid solutions in equilibrium with the resin Dowex 50-8X have been plotted versus $X_R$ in Fig. 2. The results of experiments 2A to 3H and experiments 157-1 to 157-7 are in good agreement although different batches of Dowex 50-8X and somewhat different procedures were employed. In general the behavior of both the lignin sulphonate and the sulphurous acid solutions is about the same and mostly as expected when the ammonium-hydrogen selectivity coefficient, $K = 1.2$ as found by Bauman and Eichhorn (*5*). However in the region of about $X_R = 0.7$ to 1.0, a substantially lower value of the selectivity coefficient is indicated and this "azeotrope" type situation has been observed before by Reichenberg, Pepper and McCauley (*12*) and by Gregor and Bregman (*13*).

Values found for equilibrium distribution of ammonia with other resins have been plotted in Fig. 3 and these results are in general agreement with those corresponding to an ammonium-hydrogen selectivity coefficient of about one for the Amberlite IR 100 resin and slightly lower values for the other resins, although in these cases also departures are observed in the region of $X_R = 0.70$ to 1.0. Roughly similar behavior is observed for all resins studied except the phosphonium resin which is apparently considerably less than completely dissociated under the conditions studied.

A few measurements were obtained for the process of exchange of calcium-hydrogen ions between Dowex 50-8X and sulphite spent liquor or sulphurous acid solutions:

$$Ca_S^{++} + 2H_R^+ \leftrightharpoons Ca_R^{++} + 2H_S^+ \quad (19)$$

and results are given in Table III. These values were correlated using the relationship:

$$\frac{X_R}{(1 - 2X_R)^2} = K\left(\frac{C_R}{C_S}\right)\frac{X_S}{(1 - 2X_S)^2} \quad (20)$$

which was obtained by Bauman and Eichhorn (*5*). For the computations, one can assume that the following ionization reaction and equilibrium can be used:

$$(LSO_3)_2Ca \leftrightharpoons Ca^{++} + 2LSO_3^- \qquad K_{LSCa_{1/2}} = \frac{(C_{Ca})(C_{LS})^2}{(C_{LSCa_{1/2}})} \quad (21)$$

Use of the same type of development as carried out above leads to the equations:

$$C_{HSO_3}^2 + C_{HSO_3}(K_{H_2SO_3} - 2C_{Ca}) - (K_{H_2SO_3}C_{TSO_2}) = 0 \quad (22)$$

and

$$C_{LS}^4 + C_{LS}^3(K_{LSH}) - C_{LS}^2(C_{TLS}K_{LSH} - 2C_{TCa}K_{LSH} - K_{LSCa_{1/2}}) + C_{LS}(K_{LSH}K_{LSCa_{1/2}} - 2K_{LSCa_{1/2}}C_{TCa}) - (K_{LSCa_{1/2}}K_{LSH}C_{TLS}) = 0 \quad (23)$$

where the calcium concentration is expressed in moles per liter. Again, for lack of reliable dissociation constants, attempts have not been made to calculate $X_S$ for the lignin sulphonate solutions.

The values of $X_S$ found for sulphurous acid and of $X_S'$ for lignin sulphonate solutions in equilibrium with Amberlite IR 100 resin are given in Table III and the quantities $2X_S$ or $2X_S'$ are plotted versus $2X_R$ in Fig. 4. The experimental data over most of its range are roughly represented by the full line shown for $(K)$ $(C_R/C_S) = 10$ and this can be compared with the dashed line shown for $(K)$ $(C_R/C_S) = 8$ as found by Bauman and Eichhorn for calcium-hydrogen ion exchange at 0.1 $N$ total salt concentration.

In view of the relationships found in this investigation for the ammonium-hydrogen and for the calcium-hydrogen ion exchange processes, it is probable that exchange of hydrogen ions for other monovalent ions such as sodium, or for divalent ions, would proceed about as would be predicted from the selectivity coefficients for these processes as reported by Bauman and Eichhorn.

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# Sizing Paper Under Neutral or Alkaline Conditions


W. F. REYNOLDS, L. A. LUNDBERG, and J. C. BARTHEL



**A new sizing agent, the salt of a hydrophobic polyfunctional amine, has been developed to size neutral and alkaline papers, including calcium carbonate filled papers. Dissolved in hot water it produces a cationic colloid of exceedingly small particle size. The size is applicable to a wide variety of pulps. The size may also be applied at the size press or calenders to impart sizing or augment conventional sizing. Application variables involved in using the size are described with a discussion of such factors as size-fiber contact time, stock temperature, effect of various anions and alkaline fillers, stock pH, presence of rosin size and alum, and drying conditions. Among the outstanding properties imparted by the size to paper are high resistance to the penetration of writing ink and alkaline solutions, reduced tendency of the paper to curl when wetted on one side, improved resistance to color degradation on exposure to ultraviolet light, and improved resistance to butyl carbitol penetration.**


The conventional materials used to size paper include such well-known products as rosin, asphalt, and wax sizes used in conjunction with aluminum sulphate. The sizing of paper with rosin size dating from the early 19th century has been the subject of considerable research and controversy ever since. Wax sizes represent a much newer approach and were developed principally as an adjunct to beater rosin sizing to improve the water and lactic acid resistance of paper; surface application of wax sizes also has advanced quite rapidly.

Beater addition of these sizing agents requires acidic papermaking conditions and the addition of an aluminum salt. In the majority of instances the pH of the stock is maintained between pH 4.5 and 5.5 to obtain optimum results; a limitation which has restricted the manufacture of sized papers to acidic grades. Attempts to size alkaline papers, particularly calcium carbonate filled papers with modified rosin and wax sizes are cited in the patent literature (*3*, *4*, *5*). With calcium carbonate filled papers one procedure has been to introduce the alum to the rosin or wax sized stock as close to the wire as possible. In general, the sizing results obtained have left much to be desired.

The sizing agent to be described in this paper was selected from a number of synthetic compounds developed especially for sizing paper under alkaline conditions. Subsequent work with this compound revealed that it was operative under neutral and acidic conditions as well. This will be discussed in more detail later.

The new product* may be described as a thermoplastic, water-dispersible synthetic wax. Chemically it is a salt of a hydrophobic polyfunctional amine. It is manufactured in the form of light brown flakes containing 100% active ingredients and is easily prepared


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* Sold commercially as CYRON® Size.

for use by dissolving in hot water at concentrations up to 5% solids. A temperature of 165 to 175°F. is sufficient to completely dissolve the material in 30 min. with agitation. Where hard water conditions (carbonate hardness) are encountered it may be advisable to preacidify the water with acetic acid to a pH of 4.5. The solution is stable. The solution of this material cannot be classed as a true solution but rather as a colloid of exceedingly small particle size possessing a cationic charge.

The application of the new sizing agent to paper will be divided into two categories for discussion: slush stock (beater) treatment and surface treatment (size press or calender).

## SLUSH STOCK TREATMENT

The successful application of the new sizing agent to paper stock is attributed to its cationic nature. It is very effectively retained by papermaking fibers when used properly.

In contrast to rosin sizing where difficulty is experienced with highly purified fibers (*1*), this new way of sizing paper is applicable to practically any type of fiber ranging from groundwood to alpha pulp or rag-stock. It is true that somewhat larger quantities, up to 3% based on dry fiber, are required with the purified fibers. In general, about 0.25 to 2.0% size (based on dry weight of the fiber) is adequate, depending on the type of fiber, water conditions, degree of sizing required, and other factors. In this connection it was found that there is generally a rather definite threshold amount, below which no sizing is apparent, and above which satisfactory sizing is obtained. Amounts of size greater than the threshold value will give even higher sizing values.

### *Addition Variables*

In applying any new material to stock the question arises as to where it should be added to the stock and under what conditions. The factors that have an influence on this decision are listed below with a brief explanation of each. They are not necessarily in the order of their importance:

*Effect of Refining the Sized Fiber.* The extent to which refining exerts a detrimental influence on the effectiveness of a stock treatment largely depends on the ability of the additive to be redeposited on the freshly exposed fiber surfaces. In the case of this new sizing material the treated stock may be subjected to moderate refining without impairing the sizing provided the stock is not refined under strongly alkaline conditions.

**Table I. Effect of Stock pH at Point of Synthetic Size Addition**

Sulphite-soda blend (60–40) furnish

| *Material used for adjustment of stock pH prior to size addition* | *Stock pH before size addition* | *pH of size treated stock*[a,b] | *Water resistance,*[c] *sec.* | *Ink resistance,*[d] *sec.* |
|---|---|---|---|---|
| HCl | 4.9 | 4.5 | 50 | > 600 |
| NaOH | 7.7 | 6.4 | 47 | > 600 |
| NaOH | 8.3 | 7.2 | 42 | > 600 |
| NaOH | 8.6 | 7.5 | 47 | > 600 |
| CaO | 8.9 | 7.7 | 41 | > 600 |
| NaOH | 8.9 | 7.9 | 45 | > 600 |
| $Na_2SiO_3$ | 8.9 | 8.6 | 39 | > 600 |
| $NaAlO_2$ | 8.9 | 8.75 | 37 | > 600 |
| $Na_2CO_3$ | 8.9 | 8.75 | 35 | > 600 |
| NaOH | 9.0 | 8.9 | 34 | > 600 |
| NaOH | 9.2 | 9.2 | 24 | > 600 |
| NaOH | 9.5 | 9.5 | 17 | > 600 |
| NaOH | 9.8 | 9.8 | 11 | > 600 |

[a] Stock consistency was 0.6%.
[b] Size treated fiber was adjusted to pH 9.0 with NaOH or HCl as required and sheets formed at pH 9.0.
[c] Currier size tester—slack scale.
[d] Barss, Knobel & Young news ink penetration tester using Waterman Ideal ink.

*Contact Time Between the Size and Fiber.* Due to the cationic nature of the size a fiber-size contact time of 10 sec. or possibly less is usually sufficient for development of maximum sizing properties of the finished paper.

*pH of the Stock at Point of Size Addition.* As mentioned earlier the synthetic size is effective over a wide pH range, for example, between pH values to 4 and 11. To assure maximum sizing efficiency the pH of the stock at the point of size addition should be below pH 8.5. At higher stock pH values at the point of size addition the sizing efficiency drops off. This effect is shown by the data in Table I. It should be emphasized, however, that once the size has been added under the favorable pH conditions just mentioned, the pH may be adjusted to a higher value without loss of sizing.

*Stock Temperature.* The size may be added to stock at temperatures up to 130°F. and the sheet formed at high headbox temperatures without affecting the sizing efficiency. In contrast, the efficiency of rosin size at 130°F. is rather poor (*2*).

*Effect of Anions.* The influence of anions in the stock at the point of size addition depends on the stock consistency and the specific anion involved. For example, concentrations of sulphates higher than 200 p.p.m. in 0.6% consistency stock are detrimental whereas in 6% consistency stock up to 1000 p.p.m. of sulphates can be tolerated without seriously impairing the sizing. Since alum is not necessary with this new synthetic size it is recommended that it not be used unless there is some other function, such as filler or dyestuff retention, for which it is required. In those instances where the use of alum would introduce detrimental concentrations of sulphates, much better sizing is obtained if the synthetic size is added before the alum.

Of the other anions studied, chlorides, borates, and phosphates, at 1000 p.p.m. in 0.6% consistency stock have no ill effects on the sizing efficiency under acidic conditions but do cause a slight decrease in sizing under alkaline papermaking conditions. Where an alkaline compound is required for pH adjustment of stock after treatment with the size, any of the common alkalies are satisfactory, e.g., sodium hydroxide, sodium carbonate, sodium silicate, sodium aluminate, calcium carbonate, or calcium hydroxide.

The influence of calcium carbonate filler in the stock at the point of size addition is more a function of the amount of calcium carbonate filler based on the dry fiber than the concentration of carbonate anion. This is to be expected in view of the low solubility of calcium carbonate. For example, a minimum amount of size that will satisfactorily size a given quantity of fiber and calcium carbonate cannot be expected to size an additional amount of calcium carbonate filler. For best all-around results in sizing calcium carbonate filled paper it has been found that the size should be added to thick stock before the addition of the calcium carbonate filler. However, white water containing as much as 16,000 p.p.m. calcium carbonate can be used for beater make-up provided the synthetic size is added at 5 to 6% stock consistency.

**Table II. Comparison of Rosin Size Versus Synthetic Size with Various Pulps**

| *Pulp employed* | *Stock treatment, % solids based on moisture-free fiber* | *Sheetmaking, pH* | *Water resistance,[a, c] sec.* | *Ink resistance,[b, c] sec.* |
|---|---|---|---|---|
| Unbleached sulphite | 1.25% rosin size and 2% alum | 5 | 49 | 85 |
| | 1.25% synthetic size | 5 | 34 | > 600 |
| | 1.25% synthetic size | 9 | 27 | > 600 |
| Bleached sulphite | 1.25% rosin size and 2% alum | 5 | 51 | 120 |
| | 1.25% synthetic size | 5 | 49 | > 600 |
| | 1.25% synthetic size | 9 | 48 | > 600 |
| Sulphite-soda (60–40) | 1.5% rosin size and 2% alum | 5 | 59 | 280 |
| | 1.5% rosin size and 2% alum | 9 | < 1 | < 10 |
| | 1.5% synthetic size | 5 | 58 | > 600 |
| | 1.5% synthetic size | 9 | 54 | > 600 |
| Unbleached sulphate | 1.25% rosin size and 2% alum | 5 | 42 | > 600 |
| | 1.25% synthetic size | 5 | 57 | > 600 |
| | 1.25% synthetic size | 9 | 77 | > 600 |
| Bleached sulphate | 1.25% rosin size and 2% alum | 5 | 54 | 270 |
| | 1.25% synthetic size | 5 | 36 | > 600 |
| | 1.25% synthetic size | 9 | 45 | > 600 |
| Unbleached groundwood-sulphite (75–25) | 1.25% rosin size and 2% alum | 5 | 110 | 250 |
| | 1.25% synthetic size | 5 | 94 | > 600 |
| | 1.25% synthetic size | 9 | 91 | > 600 |
| 100% rag | 2.5% rosin size and 2% alum | 5 | 12 | 65 |
| | 2.5% synthetic size | 5 | 29 | > 600 |
| | 2.5% synthetic size | 9 | 21 | 520 |

[a] Currier size tester on slack scale.
[b] Modified Barss, Knobel & Young news ink penetration tester using Waterman Ideal ink.
[c] The water and ink resistance tests were made on sheets averaging 47 lb. basis weight (25 × 40—500).

*Use with Rosin Size.* The effect of rosin size on the sizing efficiency of the new synthetic size depends on a number of factors which will be touched on briefly. Where alum is present, rosin size does not appear to affect the efficiency of the synthetic size unless the paper is formed under alkaline conditions. In the absence of alum, however, a small amount of rosin size can be detrimental. As a result of the study of the interaction of rosin size, alum, and the synthetic size two rather interesting phenomena were noted. In the case where rosin size alum, and synthetic size are all present in the slush stock the sizing imparted to the finished paper is a sole function of the amount of synthetic size used. The rosin size and alum play no part at all in the sizing obtained. Furthermore, although this paper may exhibit good resistance to water and writing ink, it will not possess the resistance to 2% NaOH solution that the synthetic size is known to impart to paper.

Thus, from the above discussion, it is indicated that rosin size and alum contribute nothing to the effectiveness of the synthetic size and in some applications would be harmful. However, many factors remain to be investigated and it is quite possible that results may vary according to local papermaking conditions.

*Paper Drying Conditions.* As far as paper machine operation is concerned one of the things to consider is the drying of the paper.

Fortunately full development of the sizing is realized under the normal machine drying conditions. Under laboratory conditions, however, where sheets are sometimes dried at room temperature, no sizing is developed unless the sheets are given a heat treatment, e.g. in an oven for a minute or two at 220°F. or higher. Additional heating of the paper does not seem to have any further effect on the sizing.

## SURFACE TREATMENT

The size can be applied with or without starch or glue to the surface of sized or unsized paper. For surface treatment the acidic size solution can be neutralized, if desired, with sodium hydroxide or ammonia to a slightly alkaline pH with no appreciable increase in viscosity. The size is not compatible with some of the converted starches and compatibility tests should be run on laboratory scale before making full scale mill trials. When used with the synthetic size, chlorinated starches have a greater tendency than the enzyme-converted starches to give high viscosity solutions.

If air is introduced into the size solution or starch solution containing the size, foaming may take place with neutralized solutions when the temperature is in a specific range. Up to 115°F. the foam is of a light nature and can be easily broken up. Between 115 to 160°F. the foam is more persistent. This condition can be eliminated by the addition of extremely small

**Table III. Resistance of Synthetic Sized and Rosin Sized Sheets to Various Liquids**

| | | | *Absorptiveness (Cobb test) TAPPI 441 m-45 expressed as grams solution absorbed per 100 sq. cm. at 73°F. and 50% R.H.[a]* | | | | |
|---|---|---|---|---|---|---|---|
| *Pulp employed* | *Stock treatment, % solids based on moisture-free fiber* | *Sheet-making pH* | *1% lactic acid for 60 min. (pH 2.5)* | *Distilled water for 60 min. (pH 6.5)* | *1% $NaHCO_3$ solution 60 min. (pH 8.25)* | *1% $Na_2B_4O_7$ solution for 60 min. (pH 9.1)* | *1% $Na_2CO_3$ solution for 60 min. (pH 11.2)* | *2% NaOH solution for 15 min. (pH 13.1)* |
| Bleached sulphite | 1.25% rosin size and 2% alum | 5 | .. | 0.91 | .. | .. | .. | 4.80[b] |
| | 1.25% synthetic size | 5 | .. | 0.85 | .. | .. | .. | 1.65[c] |
| | 1.25% synthetic size | 9 | .. | 0.96 | .. | .. | .. | 1.15[c] |
| Bleached sulphite-soda (60–40) | 1.5% rosin size and 2% alum | 5 | 1.57 | 1.06 | 1.48 | 1.28 | 3.06 | .. |
| | 1.5% synthetic size | 5 | 1.15 | 0.84 | 0.85 | 0.82 | 0.72 | |
| | 1.5% synthetic size | 9 | 0.86 | 0.80 | 0.88 | 0.80 | 0.76 | |

[a] The Cobb tests were made on 200 lb. (25 × 40—500) basis weight handsheets.
[b] The 2% NaOH solution penetrated the sheet in less than 1 min.
[c] The 2% NaOH solution did not penetrate the sheet.

**Table IV. Aging Characteristics of Paper**

| Bleached sulphite pulp treatment, % solids based on moisture-free fiber | Sheet-making, pH | Brightness, % reflectance at 457 mmu | | | |
|---|---|---|---|---|---|
| | | Initial | Heat exposure[a] | Ultraviolet exposure[b] | Fadeometer exposure[c] |
| None | 5 | 84.2 | 79.6 | 77.9 | 81.3 |
| None | 9 | 83.9 | 80.1 | 78.2 | 81 7 |
| 1.5% rosin size and 2% alum | 5 | 79.5 | 72.3 | 72.2 | 73.6 |
| 1.5% synthetic size | 5 | 79.0 | 71.8 | 76.8 | 82.5 |
| 1.5% synthetic size | 7 | 79.2 | 72.4 | 78.0 | 83.4 |
| 1.5% synthetic size | 9 | 79.1 | 72.9 | 78.3 | 83.4 |

[a] Sixteen hours in forced draft oven at 220°F.
[b] Fifteen-minute exposure.
[c] Sixteen-hour exposure.

amounts of a silicone defoamer. Above 160°F. very little foaming is encountered.

## EFFECT OF SIZE ON PROPERTIES OF PAPER

The ability of the new synthetic size to size alkaline papers makes it now possible to produce grades heretofore not considered. Indeed, judging by the literature very little work has ever been done to study the advantages of making paper under alkaline conditions. Among the few advantages that have been mentioned are improved permanence and noncorrosiveness toward metals. Future investigations will undoubtedly uncover many more.

The type of sizing imparted by the synthetic size appears to be quite different from that obtained with rosin size. To begin with the behavior of water on the surface of the sheet is very different from that on the rosin sized paper. The contact angle is much lower on the special sized paper yet the internal penetration of the liquid is generally much less. This phenomenon has been taken advantage of in coating raw stock where very rapid and uniform wetting of the surface accompanied by controlled penetration of the coating into the sheet is very desirable.

Paper sized with the synthetic size exhibits much greater resistance to writing inks than paper sized with rosin size. Indeed, the paper will float on the ink for days without ink striking through the paper. Excellent pen and ink writing characteristics are imparted by the size with no feathering tendency noted. A comparison of sizing results obtained with rosin size and the synthetic size on a variety of pulps is shown in Table II.

One of the most remarkable properties imparted to paper by the new sizing material is resistance to alkalies. The sized paper is not only resistant to solutions of such alkaline substances as soda ash, sodium silicate, sodium phosphate and other alkaline salts but is also resistant to dilute solutions of caustic soda. Furthermore, the sized paper may be exposed to alkaline liquids, dried, and still exhibit good sizing. Test data illustrating the resistance to various liquids are shown in Table III.

Compared with rosin sized paper, paper sized with the synthetic size exhibits very little tendency to curl when the paper is wetted on one side. Apparently the unequal stresses that cause rosin sized paper to curl when moistened on one side are not set up in paper sized with the new size.

Good success has been attained in sizing very lightweight paper, e.g., individual plies of a wadding paper. This probably can be attributed to the high degree of retention of the size by the paper fibers and complete coverage of the most minute fibrils by the exceedingly tiny cationic size particles. Electron micrographs of rosin sized and synthetic sized fibers definitely support this explanation.

In many instances paper sized with the synthetic size has exhibited lower strength properties, indicative of the fact that the size has interfered with normal fiber bonding forces. The extent to which this weakening effect prevails depends on the type of fiber used and the amount of size employed. Generally speaking the weaker pulps, such as groundwood and short-fibered pulps, are not affected as much. The softening effect associated with a loss in strength properties has been found advantageous where sized paper possessing improved softness is desired. A number of instances has been cited where no strength loss was noted, thus making it advisable to consider each case individually. The use of cooked starch and other dry strength additives has proved to be an effective way of minimizing this effect.

Comparative data showing the effects of the synthetic size and rosin size on the brightness of the sized paper before and after accelerating aging tests are presented in Table IV. The synthetic size and rosin size appear to have about the same effect on the initial brightness of sized paper and after a 16-hr. heat treatment.

On exposure to ultraviolet light synthetic sized paper exhibits much greater resistance to color degradation than the rosin sized paper. Exposure in a Fadeometer machine shows even more striking difference between the two kinds of sizing materials, the brightness of the synthetic sized paper shows a marked improvement whereas the rosin sized paper and even unsized paper suffer a drop in brightness.

Paper or paperboard internally or surface sized with the synthetic size exhibits excellent resistance to butyl carbitol, a test that reportedly correlates with the resistance of paper to printing inks. In actual practice the synthetic size has greatly improved the printability in some instances and in others has had no effect.

## COMMERCIAL APPLICATIONS

The new size has been found useful in a wide variety of papers, including such grades as noncorrosive wadding, napkin, coating rawstock, specialty bag, hanging, carbonate filled text, specialty board, label, and alkali resistant papers.

It is hoped that this unique sizing material will further assist papermakers in the development of new paper grades and specialties, and provide another tool for imparting a wider variety of desirable use properties to paper.

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# An Investigation of Haze in Cellulose Acetates Made from Wood Pulps

KEITH E. BRADWAY

**A study was made of the "haze"—i.e., that substance which caused turbidity in acetone solutions—isolated from three cellulose acetates made from wood pulps and one from a linter acetate. Small amounts of haze could be removed by centrifugal means, even from commercially suitable acetates. The coarsest fraction always contained some fibers or fiber fragments; otherwise the hazes consisted of amorphous masses. The hazes from commercial acetates had low acetyl contents, whereas those from two very hazy acetates had acetyl contents in the range of acetone solubility. Despite their acetyl content the hazes were insoluble except in formic acid. The haze from a very hazy acetate was richer in ash than the remainder of the acetate but contained predominantly polysaccharides. Hazes from wood pulp acetates always contained xylans and mannans with large amounts of cellulose. All the haze was removed by fractional precipitation from two cellulose acetates made from wood pulp. All of the mannans and xylans originally in the acetates were present in the total hazy fractions, and apparently were one cause of the haze. The hazy fraction from a very hazy acetate caused filtration difficulties, was higher in combined sulphate, uronic acids, and ash, and contained particles which caused thick spots in a film. This fraction was not solubilized by reacetylation, but was clarified if reacetylated after removal of mannan and xylan. Cellulose probably was present in the haze because it was shielded from reaction.**

Cellulose acetate requires the use of high quality celluloses for its manufacture; until recently cotton linters were the only fibrous raw materials being used. The wood pulps which to a large extent, are now used, yield acetates whose solutions are generally turbid or hazy. This haze causes difficulty in filtration and detracts considerably from the clarity of films. An understanding of the constitution and the origins of the haze is of considerable commercial importance.

In general two qualities affect the suitability of pulps for acetylation, notably, purity and reactivity. The only acceptable test for the suitability of pulps is an actual acetylation. Procedures have been developed utilizing a test acetylation and evaluation of the primary acetate by the turbidity, amount of insoluble material, and filterability.

It is generally assumed that hemicelluloses cause haze. Jayme and Schenk (*1*) found a positive correlation between the pentosan and mannan contents of pulps and the turbidity of the primary acetates formed from them, but Jayme (*2*) maintained that xylan, mannan, and holocellulose can be acetylated to form soluble products. Jayme (*3*) found that straw xylan extracted by hot alkali yielded an acetate soluble in acetic acid, whereas a xylan extracted by cold alkali yielded a very turbid acetate. Whistler (*4*) stated that xylan diacetates are insoluble at high molecular weights but become increasingly soluble when degraded. Sobue and Fujimura (*5*) found that a beech xylan was more reactive than cellulose but yielded an insoluble diacetate. Millet and Stamm (*6*) and Wethern (*7*) acetylated isolated hemicelluloses and obtained insoluble products.

Pringsheim and Liss (*8*) acetylated salep mannan to a triacetate which was soluble in chloroform and acidic acid. Patterson (*9*) and Klages and Niemann (*10*) have acetylated ivory nut mannans to the triacetate stage. The products were soluble in chloroform, although Patterson noted a tendency for the triacetate to gel in chloroform.

Howlett and Urquhart (*11*) maintained that combined sulphate in large amounts may cause haze, and Engelmann (*12*) stated that uronic acids and carboxyl groups cause haze. These groups are generally believed to cause cross-linking by means of divalent cations, particularly calcium. Despite this, MacClaren (*13*) acetylated pulps that had been washed with distilled water to exclude calcium ions and obtained acetates whose solutions were more turbid than the acetates produced from pulps containing calcium ions.

The activity of celluloses is known to be decreased by the treatments necessary to remove impurities (*14*). Staudinger, *et al.* (*15*) stated that natural celluloses are semiactive, whereas mercerized or regenerated celluloses are inactive but become highly active after suitable treatment. Centola and Pancirolli (*16*) acetylated an artificially cross-linked cellulose and found it unreactive. Jayme and Schenk (*1*) believed that poorly reactive regions lead to undispersed portions of the acetates.

Haas (*17*) observed that insoluble substances were formed from the inner layer of the secondary wall during acetylation.

By means of fractional precipitation Sookne, *et al.* (*18*) and Engelmann (*12*) have isolated the haze from cellulose acetate solutions. Engelmann found that the most insoluble fraction of cellulose acetates from various sources contained the major portion of uronic acids, carboxyl groups, pentosans, and ash, and had a higher percentage of calcium in the ash.

## PRESENTATION OF THE PROBLEM

With the exception of Englemann's work (*12*) there has been no thorough study of the haze, and his work concerned the least-soluble fraction which probably also contained clear cellulose acetate. The isolation and study of the haze are desirable as a means of clarifying the causes of haze and the manner in which it is formed.

This investigation had as its goal the removal and characterization of haze from wood pulp acetates. Although haze is significant as a cause of filtration diffi-

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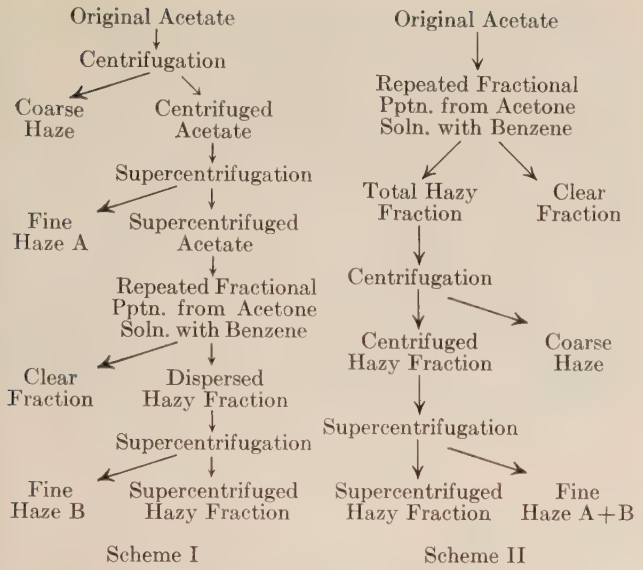


***Fig. 1. Isolation of haze from cellulose acetate***

culties, filtration was not used as a means of isolation. Instead the haze was defined as any substance that could be removed by centrifuging from acetone solution, and as any material that imparted a high turbidity to an acetone solution. Turbidities at concentrations* of 1, 2, or 4% were determined with the transparency meter as described by Heuser, *et al.* (*19*).

## RAW MATERIALS

Four acetate samples were obtained as starting materials. Two were commercial acetates, one of which was a "linter acetate" and the other a "pulp acetate" made from Rayaceta wood pulp. The other two samples, designated respectively in this study as "experimental acetates I and II," were prepared in the laboratories of the du Pont Co. by conventional methods from Rayacord wood pulp (93.6% alpha-cellulose). Experimental acetate II was purposely degraded more than was experimental acetate I during preparation and had a higher clarity and filterability and a lower amount of undispersed material. The investigation was concerned primarily with experimental acetate II.

## ISOLATION OF HAZE

Reference is made to the flow charts shown in Fig. 1. The following procedure, the first of two to be described, is termed scheme I.

A "coarse haze" fraction was removed from the acetate sample by centrifuging a 4% acetone solution for 15 min. at an average acceleration of $5.7 \times 10^5$ cm. per sec.$^2$. The coarse haze was washed three times with acetone in the centrifuge bottle and the washings were combined with the decanted solution. The centrifuged acetate solution was diluted to 2.5% concentration and centrifuged in the Sharples supercentrifuge at an acceleration at the rotor wall of $1.3 \times 10^7$ cm. per sec.$^2$. A "fine haze A" fraction was removed from the rotor and was washed by suspending in acetone and supercentrifuging again. The remainder of the haze was removed from the supercentrifuged acetate solution by fractional precipitation from the acetone solution with benzene. Nine hundred and fifty milliliters of benzene were added with stirring to 900 ml. of the supercentrifuged acetate at 5% concentration in acetone. The precipitate which formed was dissolved by heating to 50°C.; on standing at room temperature a precipitate again separated, the clear supernatant solution was decanted, and the precipitate was dissolved in acetone and refractionated several times. The clear solution which was removed by each refractionation was added to the clear fraction. The criterion for complete precipitation in the refractionation steps was the absence of turbidity in the clear fraction. The fraction which precipitated was designated the "dispersed hazy fraction" and the combined soluble fractions were designated the "clear fraction."

A "fine haze B" fraction was removed by supercentrifuging the "dispersed hazy fraction" at 0.5% concentration in acetone.

From the above procedure a slightly different method (scheme II) was evolved in which a "total hazy fraction" was first separated from the clear fraction by repeated fractional precipitation from acetone by benzene. "Coarse haze" was removed from the total hazy fraction by centrifuging as before, and a "fine haze A + B" fraction was removed by supercentrifuging a 0.5% solution of the centrifuged hazy fraction.

The clear fraction from experimental acetate II had a turbidity of 3.2% at 4% concentration compared to 5.0% for the centrifuged linter acetate. Qualitative filterability tests showed that the clear fraction caused no difficulty in filtration, whereas the total hazy fraction was very difficult to filter. Substantially all the haze was removed by this fractionation.

The two schemes of isolation were compared in their application to experimental acetate II. The yields of the various haze fractions and the turbidities of the supercentrifuged hazy fractions are compared in Table I. The comparison shows that the schemes gave very similar results; consequently, the coarse hazes and the supercentrifuged hazy fractions were considered equivalent, and the fine haze A + B in scheme II was considered equivalent to the sum of fine haze A and fine haze B in scheme I.

The acetate samples were compared by their turbidities and yields of coarse haze and fine haze A. The yields of the dispersed hazy fraction were compared for the pulp acetate and experimental acetate II. These values are shown in Table II. All the acetates contained material which could be removed by these methods.

## PHYSICAL EXAMINATION

The coarse hazes from each acetate contained some fibers and fiber fragments. The fine haze A and coarse haze from experimental acetate II were examined under the microscope and found to consist predominantly of

**Table I. Comparison of Isolation Methods**

| | *Scheme I* | *Scheme II* |
|---|---|---|
| Turbidity, %, at 1% concentration of supercentrifuged hazy fraction | 17.7 | 18.8 |
| Yield, %, based on original acetate | | |
| Coarse haze | 0.3 | 0.2 |
| Fine haze A | 1.9 | .. |
| Fine haze B | 0.7 | .. |
| Fine haze A + B | 2.6 | 2.4 |
| Supercentrifuged hazy fraction | 16.7 | 16.5 |
| Total hazy fraction | 19.6 | 19.1 |

* Concentrations of polymer solutions are given here in percentages where 1% = 1 gram per 100 ml.

Table II. Comparison of Acetate Samples

| | Linter acetate | Pulp acetate | Experimental acetates I | II |
|---|---|---|---|---|
| Turbidity, %, at 2% concentration | | | | |
| Centrifuged acetate | 2.9 | 4.3 | 24.2 | 25.3 |
| Supercentrifuged acetate | 2.3 | 3.1 | 9.2 | 13.8 |
| Yields of haze, %, based on the original acetate | | | | |
| Coarse haze | 0.1[a] | 0.15[a] | 0.5 | 0.3[b] |
| Fine haze A | 0.3 | 0.3[a] | 1.4 | 1.9[b] |
| Dispersed hazy fraction | .. | 13.5 | .. | 17.4 |

[a] Average of two determinations.
[b] Average of five determinations.

amorphous particles although the coarse fraction contained some fibers as mentioned above. According to Blume (*20*), however, these unacetylated fibers cause no filtration difficulties.

Films of about 0.15 mu thickness were formed from the centrifuged hazy fraction of experimental acetate II and examined with the electron microscope. The electron micrographs of films of two thicknesses are shown in Figs. 2 and 3. These films contained thickened areas of about 0.1 mu diameter; the connecting areas were broken up completely in the thinner film of Fig. 3. A film formed from the clear fraction did not present this type of mottled appearance.

The fine haze A and fine haze B from experimental acetate II could not be redispersed in acetone and were insoluble in cellulose acetate solvents. They were dissolved by formic acid.

The refractive indexes of films of the experimental acetate II, the dispersed hazy fraction, and the fine haze A + B from that acetate were measured using the method given by Bauer (*21*). The averages of four measurements, made with white light at 25°C., are summarized in Table III. The refractive indexes of the fine haze A + B and of the dispersed hazy fraction were not significantly different from that of the original acetate. This indicated that the effect of haze in causing turbidity could not be ascribed to differences in refractive index; therefore the film turbidity must be caused by poor drying tendencies or variations in thickness.

## CHEMICAL ANALYSES OF HAZE

### *Acetyl Content*

The acetyl contents of haze fractions were made by a semimicro Eberstadt method. One-tenth gram samples of known moisture content were swollen with 10 ml. of 75% ethyl alcohol and saponified with 10 ml. of 0.5 *N* alkali. After 24 hr. the suspension was neutralized with 10 ml. of 0.5 *N* acid, allowed to equilibrate 1 hr., and back-titrated with 0.1 *N* base. Acetyl contents of acetate fractions are shown in Table IV. In most cases the results are averages of at least two determinations. All haze fractions were acetylated appreciably.

*Fig. 2. Film of centrifuged hazy fraction of experimental acetate II (35,000 ×)*

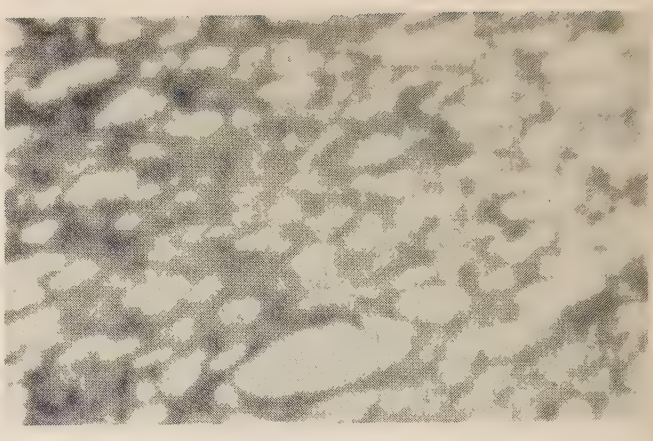

*Fig. 3. Thin film of centrifuged hazy fraction of experimental acetate II (35,000×)*

### *Carbohydrate Composition*

The proportions of simple sugars in polysaccharides were determined by hydrolysis followed by quantitative paper partition chromatography. One-tenth gram samples were dissolved with 1 ml. of cold 72% sulphuric acid, diluted to 35 ml., and boiled 4 hr. The hydrolyzate was neutralized with barium carbonate, filtered, and concentrated to 0.5 ml.

The use of paper partition chromatography for identification of sugars in pulp hydrolyzates as used here has been described by Wise, *et al.* (*22*). The hydrolyzates were first chromatographed with a butanol, pyridine, and water solvent mixture (10:3:3) and the sugar spots were made visible by spraying with aniline hydrogen phthalate solution. The spots were identified by comparison with known sugars.

Quantitative sugar determinations were made by the method of Hirst and Jones (*23*) which utilizes the quantitative oxidation of the sugars to formic acid with sodium periodate. Quantitative chromatograms were prepared by drawing a line of the hydrolyzate by means of a capillary tube across the upper part of a 4 by 24-in. strip of Whatman no. 1 filter paper. The chromatograms were developed for 60 to 72 hr. for adequate separation of the sugar bands. The bands were located by spraying a 1-cm. wide guide strip which had been cut from the middle of the paper. The zones containing the sugars were cut out and eluted with water by extraction with four 1.5 to 2-ml. portions of water. This extraction was carried out in a 6 by 1-cm. glass

Table III. Refractive Indexes of Films of Fractions from Experimental Acetate II

| Fraction | Refractive index |
|---|---|
| Original acetate | 1.493 |
| Dispersed hazy fraction | 1.497 |
| Fine haze A + B | |
| Cast from HCOOH solution | 1.495 |
| Cast from $CHCl_3$ and MeOH suspension | 1.498 |

**Table IV. Acetyl Contents of Various Cellulose Acetate Fractions**

| *Sample* | *Acetyl content, %* |
|---|---|
| Linter acetate | |
| Original acetate | 40.1 |
| Coarse haze | 28 |
| Fine haze A | 32[a] |
| Pulp acetate | |
| Original acetate | 39.9 |
| Coarse haze | 30 |
| Fine haze A | 35.9 |
| Experimental acetate I | |
| Original acetate | 39.9 |
| Coarse haze | 40.2 |
| Fine haze A | 41 |
| Experimental acetate II | |
| Original acetate | 40.0 |
| Coarse haze | 37.4 |
| Fine haze A | 39.3 |
| Dispersed hazy fraction | 39.1 |
| Fine haze A + B | 40.2 |

[a] This represents a single determination.

cylinder with a stopcock attached, into which the rolled cylinder of paper was inserted. The eluate was drained into a 15 to 20-cm. oxidation tube with a 29/42 ground glass stopper. The sugar was oxidized by heating on the steam bath for 10 min. with 1 ml. of 0.25 *M* sodium periodate. The tube was then cooled under the tap, the excess periodate was reduced with 10 drops of ethylene glycol, and the formic acid liberated from the sugars was titrated with about 0.003 *N* sodium hydroxide until yellow to methyl red indicator. A blank was subtracted from the volume and the corrected volume for xylose was adjusted by dividing by 0.96 (the ratio between the equivalent weights of pentoses and hexoses). These values were then proportional to the percentages of each sugar in the hydrolyzate.

The method was checked on known mixtures containing approximately 80% glucose, 10% mannose, and 10% xylose and checked to 2% of the sugar present. The hydrolysis procedure was shown to involve little or no loss of sugars by subjecting a known mixture of sugars to the usual hydrolysis technique.

When hydrolyzates appeared to contain only glucose, the hydrolyzate was chromatographed in the manner used for quantitative analysis. The greatest proportions of the glucose zones were cut out and discarded, the remainder of the chromatograms were then eluted, and the eluate was evaporated and rechromatographed. In this way sugars could be concentrated and detected which were present originally in amounts of less than 0.5 % of the glucose present.

Various fractions of the acetates were analyzed for their constituent sugars. These analyses are summarized in Table V. For the most part these results are the averages of four determinations. Qualitative results are included when quantitative determinations were not made.

The analyses of fractions from experimental acetate II showed that the ratios of xylose to glucose and of mannose to glucose increased with the degree of dispersion of the haze. This suggested that even more greatly dispersed haze might consist only of mannans and xylans.

The actual percentages of the various anhydrosugars present in the coarse haze and fine haze A from experimental acetate II were determined by quantitative chromatographic analyses of weighed samples to which known amounts of a reference sugar, rhamnose, were added. The haze samples were saponified before analysis. The coarse haze analyzed 94% glucose, 6% mannose, and 1% xylose; the fine haze A analyzed 67% glucose, 6% mannose, and 2% xylose. This indicated that these haze fractions were predominantly carbohydrates. The reason for the low yield of carbohydrate material from fine haze A was not investigated.

### *Identification of Sugars*

The presence of mannose and xylose was confirmed by forming chemical derivatives of 10 to 15 mg. of each sugar which had previously been isolated from the hydrolyzate of the total hazy fraction of experimental acetate II. The sugars were separated on quantitative chromatograms, eluted with methyl alcohol, and evaporated to dryness.

The mannose was precipitated with 1 ml. of phenylhydrazine reagent as the typical D-mannose phenylhydrazone. The derivative melted with decomposition at 195°C. The melting point given in the literature is 199 to 200°C.

The xylose was redissolved in methanol and evaporated to dryness again, and the dimethyl acetal dibenzylidene derivative of D-xylose was prepared according to the procedure of Breddy and Jones (*24*). The recrystallized derivative melted at 210°C.; the melting point according to Breddy and Jones is 211°C.

### *Combined Sulphate Content*

The combined sulphate contents were determined on the original experimental acetate II, on the total hazy fraction, and on the clear fraction isolated therefrom. The determinations were made according to the procedure of Malm and Tanghe (*25*). Results of duplicate determinations are tabulated in Table VI. The combined sulphate content should be higher in the haze inasmuch as the latter is less accessible to the hydrolyzing medium which causes the removal of combined sulphate.

### *Uronic Acids*

Uronic acid determinations were made by the Insti-

**Table V. Carbohydrate Analyses of Various Cellulose Acetate Fractions**

| *Sample* | *Percentage of total sugars in the hydrolyzate* | | |
|---|---|---|---|
| | *Glucose* | *Mannose* | *Xylose* |
| Linter acetate | | | |
| Coarse haze[a] | 92 | 0 | 8 |
| Fine haze A[a] | 100 | 0 | 0 |
| Pulp acetate | | | |
| Coarse haze[a] | Large | Trace | Trace |
| Fine haze A[a] | Large | Trace | Trace |
| Dispersed hazy fraction | 93 | 4 | 3 |
| Experimental acetate I | | | |
| Original acetate[a] | 96 | 2 | 2 |
| Coarse haze[a] | Large | Small | Small |
| Fine haze A[a] | Large | Small | Small |
| Experimental acetate II | | | |
| Original acetate[a] | 96 | 2 | 2 |
| Coarse haze | 94 | 4 | 2 |
| Fine haze A | 84 | 10 | 6 |
| Supercentrifuged acetate[a] | 95 | 3 | 2 |
| Dispersed hazy fraction | 88 | 5 | 7 |
| Clear fraction | 100 | 0 | 0 |
| Total hazy fraction | 88 | 6 | 6 |
| Fine haze B | 50 | 9 | 41 |

[a] These analyses were made on materials after saponification.

Table VI. Combined Sulphate and Uronic Anhydride Contents of Fractions of Experimental Acetate II

| Fraction | Combined sulphate, % | Uronic anhydride, % |
|---|---|---|
| Original acetate | 0.033 | 1.35 |
| Dispersed hazy fraction | .. | 1.4 |
| Total hazy fraction | 0.08 | 1.9 |
| Clear fraction | 0.03 | 1.2 |

tute Analytical Department and by Mrs. Ruth Rollinson of the Institute. The procedure consisted of decarboxylation with 12% hydrochloric acid and weighing of the evolved carbon dioxide according to the procedure given by Browning (*26*). Duplicate determinations on experimental acetate II and the dispersed hazy fraction, total hazy fraction, and clear fraction obtained thereform are given in Table VI. These results apparently show that the "uronic acids" were concentrated in the hazy fraction, corroborating the findings of Engelmann (*12*). However, naphthoresorcinol tests for uronic acids were negative, and no uronic acids were found by purely chromatographic means. It should be recalled that according to Browning (*27*) evolution of carbon dioxide is not an unequivocal qualitative test for uronic acids where the amount of uronic anhydride is less than 1.6%. It was certain only that the total hazy fraction contained uronic acids.

### *Ash Content*

A number of fractions of experimental acetate II were ashed; the averages of determinations are shown in Table VII. The high percentage of ash in the hazy fractions was to be expected, as ash would naturally accumulate with the material removed by centrifuging.

Spectrographic analyses of these fractions were made by Leonard Dearth of the Institute Physics Department. The major constituents of all ash fractions were calcium and magnesium; differences between the fractions were shown only in the case of minor components.

## REACETYLATION

An attempt was made to solubilize the haze by reacetylation. Thirty grams of the total hazy fraction were dissolved in 270 ml. of acetic acid and 0.54 ml. of 95% sulphuric acid. Ninety milliliters of acetic anhydride were added at 15°C., and the mixture was heated to 35°C. over a 30-min. period. The reaction was allowed to proceed 40 min. at 35°C. and stopped by adding 135 ml. of 60% acetic acid containing 1.1 ml. of 95% sulphuric acid. After 27 hr. at room temperature, the acetate was precipitated into water and washed. The coarse haze and fine haze A were isolated from this acetate. The original turbidities, haze yields, and carbohydrate compositions of the fine haze (A + B) are compared with those of the reacetylated

Table VII. Ash Content of Fractions of Experimental Acetate II

| Fraction | Ash, % |
|---|---|
| Original acetate | 0.06 |
| Coarse haze | 1.1 |
| Fine haze A | 0.8 |
| Supercentrifuged acetate | 0.06 |
| Total hazy fraction | 0.33 |
| Clear fraction | 0.05 |

Table VIII. Effect of Reacetylation on the Characteristics of the Total Hazy Fraction

| | Original hazy fraction | Reacetylated hazy fraction |
|---|---|---|
| Turbidity, %, at 1% concentration | | |
| Centrifuged hazy fraction | 52.9 | 46.9 |
| Supercentrifuged hazy fraction | 18.8 | 24.9 |
| Yield of hazy material in percentage of the original acetate | | |
| Coarse haze | 1.2 | 0.3 |
| Fine haze A + B | 12.7 | 7.6 |
| Carbohydrate composition of fine haze A + B | | |
| Glucose, % | 75 | 70 |
| Mannose, % | 9 | 11 |
| Xylose, % | 16 | 19 |

fraction in Table VIII. The small increase in the mannose to glucose and xylose to glucose ratios in the fine haze A + B indicated that cellulose had been removed from the fraction. The principal change in the total hazy fraction was that the haze was more thoroughly dispersed.

## EXTRACTION WITH SODIUM HYDROXIDE

An attempt was made to extract the mannan and xylan from the saponified total hazy fraction with 17.5% sodium hydroxide. This removed about 80% of the starting material; consequently, the material resistant to 5% sodium hydroxide was investigated. This amounted to 60% of the original saponified haze and contained 97% glucose, 2% mannose, and 1% xylose. The residue was washed, the water was displaced with acetic acid, and 15 grams of the residue were acetylated with 150 ml. of acetic acid, 0.9 ml. of concentrated sulphuric acid, and 75 ml. of acetic anhydride for $1^1/_2$ hr. at 35°C. The acetylation was stopped by adding 112 ml. of 60% acetic acid containing 0.6 ml. of concentrated sulphuric acid; the acetate was hydrolyzed for 24 hr. at room temperature and precipitated into water. The product was degraded excessively and formed a sol on attempted washing. A portion of the acetate was neutralized with sodium bicarbonate and washed with distilled water. The turbidity at 1% concentration was 21.5%, compared to 46.9% for the reacetylated hazy fraction.

## ACETOLYSIS

The fine haze A + B from experimental acetate II was acetolyzed as a means of determining whether or not cellulose was present in the haze and as a possible check on linkages between cellulose and other polysaccharides.

Five grams of the haze were added to a mixture of 28.5 ml. of acetic acid, 28.5 ml. of acetic anhydride, and 2.85 ml. of concentrated sulphuric acid at 0°C. The haze became dispersed at the end of an hour and the reaction proceeded 70 hr. at room temperature. The acetolyzate was recovered by pouring the mixture into 3 volumes of ice water containing sodium acetate, neutralizing with sodium bicarbonate, and extracting with chloroform. The very turbid chloroform extract was clarified by partially precipitating with ethyl alcohol. This yielded an insoluble fraction which, when dried, weighed 0.99 gram; the dried, chloroform-ethanol solution, when taken to dryness, weighed 4.97 grams.

The two fractions were hydrolyzed and analyzed

separately by paper partition chromatography. The insoluble fraction contained 54% glucose and 46% mannose, and the soluble material contained 78% glucose, 4% mannose, and 18% xylose. The resistance of mannan to the acetolysis may be related to the formation of haze, inasmuch as the mannan is either difficult to acetylate or else forms compounds which are so insoluble that they are inaccessible to the acetolyzing medium.

The soluble fraction was deacetylated with sodium methylate and chromatographed. The chromatogram showed large xylose, glucose, and cellobiose spots, and spots for higher polysaccharides. The presence of large amounts of cellobiose indicated that the glucose-yielding polysaccharide which was present in the haze was cellulose.

A 0.14-gram portion of the insoluble fraction was dissolved in 1.0 ml. of 72% sulphuric acid at 0°C., and acetolyzed by adding 8.8 ml. of acetic anhydride and 4.2 ml. of acetic acid. The acetolyzate was recovered as above after 70 hr., extracted with chloroform, and this extract was filtered through a bed of magnesol. One-tenth gram of acetolyzate was recovered; it was deacetylated with sodium methylate and chromatographed. Large spots for glucose and mannose were found together with four small spots in the disaccharide region. From their positions, the second disaccharide spot was cellobiose and the fourth was possibly glucosido-mannose. The disaccharides were eluted from quantitative chromatograms and hydrolyzed; the second spot yielded only glucose, the third only mannose, and the fourth yielded both glucose and mannose. If a glucosido-mannose was actually present, it would suggest that mannose units were linked to cellulose.

## DISCUSSION OF RESULTS

The separation of the total hazy fraction was accomplished by a fully described fractional precipitation on the pulp acetate and experimental acetate II (Fig. 1). The success of this method in removing all the haze was indicated by the low turbidity of the clear fraction and by the ease of filtration. Although this fraction contained all the haze, there was no simple way of showing that it did not contain clear cellulose acetate as well. The fact that the total hazy fraction from the pulp acetate amounted to 14% of the acetate, despite the low turbidity of the original acetate, indicated that the total hazy fraction probably retained some clear acetate.

The portion of the haze which was removed by centrifuging was separated from any clear acetate by washing. It was not possible, however, to remove the haze completely by centrifugal means. An increase in the amount of material which could be removed by supercentrifuging was obtained when the dispersed hazy fraction was supercentrifuged. The haze in cellulose acetates appeared to cover a wide range of particle sizes, and the fractions which were removed by centrifuging were not representative of all the haze with respect to particle size.

All the acetates contained fibers and fiber fragments. These were isolated with the coarse haze and probably represented unreactive fibers. Their presence may also have been caused by unsatisfactory mixing conditions which afforded poor contact with the acetylating medium. For the most part, however, the haze consisted of amorphous particles. The attempts to obtain sharp pictures of the haze particles with the electron microscope were unsuccessful, but the photographs at least indicated that the particles were highly swollen and caused thick spots in the films (see Figs. 2 and 3).

Both the fine hazes A and the coarse hazes from the commercial acetates had low acetyl contents indicating that their formations were caused by poor reactivity. Consequently, it may be said that poor reactivity may result either in high turbidities of acetates or in large amounts of undispersed material.

The general insolubility of the haze indicated that it was improperly acetylated, since the acetyl contents of hazes from the experimental acetates were in the range necessary for acetone solubility of cellulose acetate. The haze was probably formed as follows:

The regions of the pulp fibers, which gave rise to haze, formed insoluble acetates, at least in the outer layers. These layers were not dissolved; consequently, the acetylation did not proceed to the center of these regions. The hydrolyzing medium also failed to penetrate these layers and de-esterification occurred only at the surface.

This explains why the acetyl content of the haze did not differ markedly from that of the remainder of the acetate; the end product consisted of a mixture of acetates at varying degrees of acetylation.

The analyses of carbohydrates showed that the complete removal of the haze from experimental acetate II and the pulp acetate also caused a complete removal of mannose and xylose units. Because these substances were associated entirely with the haze, it was concluded that mannan and xylan presumably caused haze through the formation of insoluble acetates. All haze fractions from wood pulp acetates contained some mannan and xylan, and the coarse haze from the linter acetate also contained xylan. Except for the fine haze A from the linter acetate, all haze fractions contained some mannan and/or xylan.

The ash content was higher in all the haze fractions than in the clear fractions. Calcium and magnesium may have caused haze in part through cross-linking, but these elements were present in all the acetate fractions. The ash may have accompanied the haze merely because the insoluble inorganic compounds were readily removed by centrifuging and fractional precipitation methods.

Large amounts of cellulose were also invariably present in the haze. Reacetylation of the total hazy fraction after removal of the mannan and xylan resulted in a clearer acetate; thus the cellulose was able to form a soluble acetate. The combined sulphate content, although higher in the total hazy fraction than in the clear fraction was not greater than was that of soluble acetates. The uronic acids could have caused the cellulose acetate to be insoluble even though the amounts were small. No experiments were undertaken which would show that this occurred. The isolation of a polysaccharide containing both mannose and glucose units indicated that mannose units may have been present in the cellulose. This in turn might have caused cellulose to be present in the haze. It is possible, however, that the cellulose was simply shielded from reaction by mannan and xylan. If this were

true, the cellulose should have been solubilized by reacetylation, which was not the case, at least not to any great extent. On the other hand, this lack of solubilization may have been caused by the conditions of the reacetylation, the thoroughness of which is in question.

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# Effects of Mechanical Preparation and pH on the Strength of Glass-Fiber Paper*


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**Paper made entirely from glass fibers with greatly increased tensile strength, more than 300 p.s.i., was produced by changes in mechanical treatment and pH control. Beating was conducted at 85°F., and pH was adjusted to 3.5 or less. The pH was further regulated at the jordan and headbox to about 3.0. It was possible to use the jordan to break down strings in the stock, and to dry the paper to low moisture content before putting it onto the reel. The effect of beater temperature may depend on thermal expansion of the glass and decreased viscosity and interfacial tension of the water. The effect of pH is the formation of a gelatinous silica-rich layer on the fibers, which binds them together. The pH must be below 4 to obtain this effect, and the tensile strength of the paper increased as pH decreased down to 2.9. The pH of the finished paper was in the range 7.0 to 7.4.**


In the manufacture of paper from glass fibers, previously reported by the authors in *Tappi* (*1*), consideration was limited chiefly to techniques of using the paper mill equipment so as to produce a sheet of paper from glass fibers and to the effects of difference in fiber diameters and concentration of fibers in the beater on the tensile strength of the paper. The principal purpose of the work described in this report was to obtain the maximum tensile strength of glass-fiber paper without materially affecting its permeability as measured by resistance to the passage of air. To reach this goal, five factors were studied: (1) beating without lowering the roll and with different concentrations of stock in the beater; (2) beating at different temperatures; (3) the effect of the jordan on the fibers; (4) the effects of variation in the acidity, as measured by the pH value, on the fibers, and (5) the effects of different concentrations of the stock at the headbox.


Martin J. O'Leary, Superintendent of Paper Mill; Robert B. Hobbs, Chief of Paper Section; J. K. Missimer and James J. Erving, papermakers; all of National Bureau of Standards, Washington, D. C.



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### MATERIAL AND EQUIPMENT USED

The equipment and glass fibers used have been described in detail in earlier publications (*1*, *2*). In brief, two beaters were used, one of 50 lb. capacity with a copper-lined tub and manganese-bronze bars and plate, the other of 300 lb. capacity, tile-lined, with "Hytensyl" bronze bars and plate. A 29-in. fourdrinier paper machine was equipped with a 70-mesh wire 33 ft. long, one press with a rubber-covered top press roll, nine 15-in. driers, and a reel. Auxiliary equipment included a vertical stock chest with agitators moving in a horizontal plane, a jordan refiner, a 3-in. Vortrap, a flowmeter, and two Red-Ray burners over the end rolls of the drier section.

The material used for this study was Vitron AAA glass fiber, supplied by Glass Fibers, Inc. The diameters of the fibers were in the range 0.5 to 0.75 mu. A similar fiber furnished by the Owens-Corning Co. was used in preliminary experiments.

### MANUFACTURING PROCEDURES

The fibers as received were furnished to the beater in the usual manner. The concentration, times, pH values, and other conditions for the various runs are given in the tables.

**Table I. Effect of Beating Without Lowering the Roll and With Different Concentrations**

Conditions for all runs: Owens-Corning AAA fiberglas used; pH in beater 8.5; pH at headbox 8.3; concentration at headbox 0.3%

| Run no. | Beater roll setting, turns[a] | Beating time, min. | Fiber conc. in beater, % | Temperature in beater, °F. | Temperature at headbox, °F. | Basis weight (25 × 40 —500), lb. | Thickness, in. | Tensile strength Mach. dir., p.s.i. | Tensile strength Cross dir., p.s.i. | Breaking load Mach. dir., lb/in. width | Breaking load Cross dir., lb/in. width | Air resistance Mm. | Air resistance Mm./mil | Density, g./cc. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1660 | +1 | 20 | 2.0 | 72 | 90 | 42.0 | 0.0105 | 34 | 23 | 0.36 | 0.24 | 103 | 9.8 | 0.230 |
| 1705 | +1 | 15 | 1.5 | 75 | 92 | 48.4 | 0.0120 | 42 | 37 | 0.50 | 0.44 | 120 | 10.0 | 0.224 |
| 1706 | +1 | 25 | 1.0 | 72 | 92 | 45.9 | 0.0116 | 54 | 36 | 0.63 | 0.42 | 100 | 8.6 | 0.220 |
| 1708 | +1 | 35 | 0.7 | 72 | 91 | 50.6 | 0.0137 | 34 | 27 | 0.57 | 0.37 | 123 | 9.7 | 0.205 |
| 1710 | +5 | 90 | 1.0 | 74 | 90 | 46.4 | 0.0147 | 43 | 34 | 0.63 | 0.50 | 103 | 7.0 | 0.175 |
| 1712 | +5 | 90 | 1.0 | 74 | 90 | 43.8 | 0.0129 | 43 | 36 | 0.55 | 0.46 | 129 | 10.0 | 0.174 |

[a] One turn = 0.008 in.

The beater stock was dropped to the chest and pumped in a continuous stream through the stuff box and into the fan pump or the jordan. During the first few series of runs the jordan was by-passed, but in the later series the stock was given a light treatment in the jordan as shown in Tables III and IV. The stock was then propelled into the Vortrap, where the whirlpool action separated some glass pellets (about 1 mu in diameter and less) from the fibers. The pellets settled to the bottom of the Vortrap. The stock then flowed through the riffle box, by-passing the screens, into the screen hopper, and thence through the headbox onto the paper machine. The couch roll was not used. A rubber top roll was used on the first press.

In the previous article on the manufacture of glass-fiber paper it was noted that passage of this paper through rolls of which one or both surfaces were metal gave a product containing pinholes. Electron micrographs of the fiber show the presence of glass pellets, as demonstrated by Fig. 1. It is thought that the breakage of the fibers when the glass paper is passed between two close metal rolls results from the fact that the pressure forces the pellets through the paper. The removal of some of the pellets by the Vortrap reduces the tendency toward the formation of pinholes; the use of a rubber top roll acting as a cushion at the first press eliminates them. A suction couch roll would probably be very helpful in avoiding crushing of the fibers.

In the previous work it was necessary to maintain a fairly high moisture content in the glass paper in order to carry it onto the reel for the glass paper had a greater tensile strength when wet than when dry. The additional strength was sufficient to eliminate breakage between the driers and the reel. This procedure is no longer necessary. Glass papers made with the control of pH and mechanical treatment described in this article have enough strength (up to eight times that formerly attained) so that they can be dried to the lower moisture contents customarily used for cellulose paper and still be carried onto the reel without breaking.

## EFFECTS OF MECHANICAL PREPARATION OF THE FIBERS

### *Beating Without Lowering the Roll*

Two machine runs, 1710 and 1712, were made without lowering the beater roll, to determine whether the fiber bundles could be separated by circulation and also to discover whether this procedure had any detrimental effects on the fibers. The results of these runs are given in Table I, with results from four runs previously reported (runs 1660, 1705, 1706, and 1708) for comparison. It was found that a period of 90 min. in the beater was required to effect separation of the fibers. In comparing the test data with previous runs, it is seen that there was not enough improvement to warrant the long beating time. The data in the table show that the strength of the product was low. The unusually low density reported for these runs is ascribable to the fact that a new woolen jacket had been installed on the first press top roll; this jacket had not been run long enough to get rid of its original softness.

### *Effects of Beating at Different Temperatures*

Another series of runs, 1781, 1754, 1766, and 1774, was made to study the effects of different temperatures on the beating (Table II). The beater roll setting was the same for these four runs, 0.008 in. off the bedplate. A concentration of 1% fiber and a pH of 8.4 were used for all four runs. The temperatures reported were maintained within ±2°F. during the beating.

At the lowest temperature of 52°F., a period of 55 min. beating was required to disperse the fibers. As might have been expected, the tensile strength of the resulting paper was much less than that obtained in the other runs in this series, which had 50 to 100% greater strength.

When beating was done at 70°F., the fibers dispersed adequately in 25 min., and the tensile strength was increased 50% over run 1781. Again when the temperature of beating was raised to 85°F., a shorter

*Fig. 1. Showing glass pellets with fibers magnified 6600 times*

**Table II. Effect of Beating at Different Temperatures**

Conditions for all runs: Glass Fibers, Inc., Vitron AAA fibers used; beater roll setting +1; fiber concentration in beater 1.0%; concentration of stock at headbox 0.3%

| Run no. | Beating time, min. | Temperature in beater, °F. | Temperature at headbox, °F. | pH in beater | pH at headbox | Basis weight (25 × 40 —500), lb. | Thickness, in. | Tensile strength Mach. dir., p.s.i. | Tensile strength Cross dir., p.s.i. | Breaking load Mach. dir., lb./in. width | Breaking load Cross dir., lb./in. width | Air resistance Mm. | Air resistance Mm./mil | Density, g./cc. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1781 | 55 | 52 | 54 | 8.4 | 3.6 | 32.2 | 0.0075 | 81 | 54 | 0.61 | 0.41 | 99 | 13.2 | 0.238 |
| 1754 | 25 | 70 | 88 | 8.4 | 3.6 | 41.7 | 0.0090 | 128 | 82 | 1.15 | 0.74 | 118 | 13.1 | 0.257 |
| 1766 | 25 | 85 | 80 | 8.4 | 3.4 | 29.8 | 0.0075 | 177 | 110 | 1.33 | 0.83 | 110 | 14.7 | 0.220 |
| 1774 | 25 | 120 | 90 | 8.4 | 3.4 | 34.5 | 0.0075 | 152 | 93 | 1.14 | 0.70 | 105 | 14.0 | 0.254 |

beating time was required, and the tensile strength was further increased to about 100% more than resulting from beating at 52°F. A beating temperature of 120°F. gave no shortening of the beating time, and no further increase of tensile strength. Indeed, there is apparently a small decrease in tensile strength of the product as compared with run 1766, though the size of the decrease is so small that it cannot be said to be significant with any degree of confidence.

It is possible that the expansion or contraction of the fibers with higher or lower temperatures may affect the resistance of the fiber bundles to the penetration of water. Possibly also the decreased viscosity and interfacial tension of the water at higher temperatures may play a part.

From the results of these experiments, a beating temperature of 85°F. was chosen for future runs, as being effective and easy to maintain.

***Effects of the Jordan on the Fibers***

Glass fibers have some of the characteristics of rag fibers. They are "lazy" and will hang on to any projection and form lumps. This is especially true in a vertical cylindrical chest with horizontal agitators. Strings of glass fiber form on the bottom part of the agitator, fall off, and come over the machine. In a horizontal chest, the fibers are mixed better and do not form strings as they do in the vertical chest. However, some lumps do come over, possibly from the stuff pump.

The possibility of giving the stock a light treatment in the jordan in order to minimize these lumps and improve the formation was, therefore, considered even though in the previous publication it was reported that the use of the jordan produced "fines" and weakened the paper.

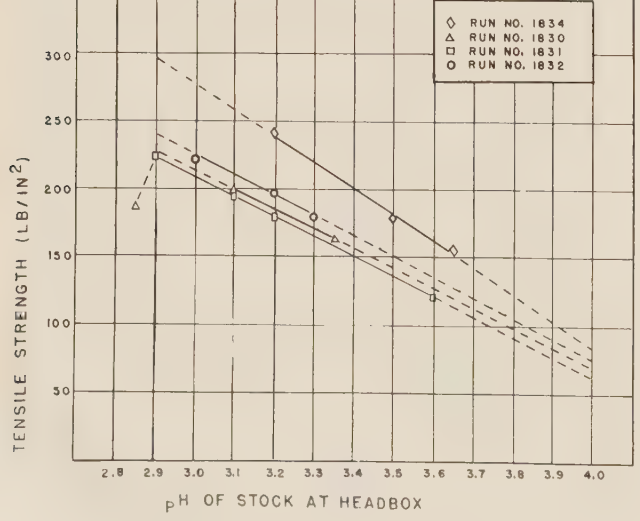


*Fig. 2. Effects of the pH on tensile strength*

This study showed that glass fiber stock that had been treated with acid, as described in the next section, could be passed through the jordan without damage or weakening of the fiber, provided the fibers are not backed up in the jordan. The acid reacted with the glass before the jordan dispersed the fibers; therefore, the jordan had no ill effect on the fibers, but it did dispose of the strings and lumps present in the stock.

The paper appeared smoother on the wire when acid was added to regulate the pH and the stock was passed through the jordan.

***Effects of Acid on the Fibers***

A series of runs, Table III, was made in which the stock in the beater was acidified with sulphuric acid to pH 3.0. (Sulphuric acid was used for all the experiments reported in this article, though other highly ionized mineral acids have been used with similar effects.) In 15 min. the fiber bundles were well dispersed and the stock was emptied to the chest. However, at the end of the beating period, the pH had risen to 3.5, and by the time the stock was ready for the machine, the pH was 4.0. In order to maintain the pH value at the desired level, further additions of acid were made. These were accomplished with constant-level reservoirs of acid, from which the solution ran out dropwise, either just before the stock entered the jordan, or at the headbox.

In the series 1831, the pH in the beater was adjusted to 3.2 initially, and rose to 3.85 as the beating progressed. Acid was later added at the jordan and headbox to secure a range of pH values from 2.9 to 3.6. The results, given in Table III and plotted in Fig. 2, show how the tensile strength of the paper increased as the hydrogen ion concentration increased, being an inverse linear function of pH value.

Series 1832 was similar to 1831 except that the percentage of long fibers was 33%, 3% higher than that of 1831. The results, again plotted on Fig. 2, show a tensile strength for the paper about 20 p.s.i. higher than 1831, for equal pH values at the headbox. As other factors than fiber length were unchanged, the increase is attributable to the increased proportion of long fibers.

In series 1830 it is observed that when the pH at the headbox was 2.85 the tensile strength was lower than when the pH was 3.1. It appears from Fig. 2 that the maximum tensile strength would be reached at pH 2.9. If the straight lines are extrapolated to pH 4.0, they reach tensile strength values from 60 to 80 p.s.i., about the expected value for paper made with a beater temperature of 85°F. and a pH of 8.4, as indicated by previous runs. The pH range 2.9 to 4.0 coincides with the findings of Hubbard and Hamilton on the durability of optical glasses (*3*). When the pH is 2.9 or

**Table III. Effect of Acid**

Conditions for all runs: Fiber concentration in beater 0.5%; concentration of stock at headbox 0.2%; beater roll setting +1; jordan setting +4 (0.045 in.)

| Run no. | Beater furnish | Beating time, min. | pH in beater | pH at headbox | Temperature in beater, °F. | Temperature at headbox, °F. | Basis weight (25 × 40—500) lb. | Thickness, in. | Tensile strength Mach. dir., p.s.i. | Tensile strength Cross dir., p.s.i. | Breaking load Mach. dir., lb./in. width | Breaking load Cross dir., lb./in. width | Air resistance Mm. | Air resistance Mm./mil | Density, g./cc. | Long fibers,[a] % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1830A | Vitron AAA | 15 | 3.3 –3.5 | 3.35 | 85 | 91 | 40.4 | 0.0095 | 165 | 89 | 1.57 | 0.84 | 100 | 10.5 | 0.236 | 32 |
| 1830B | Vitron AAA | 15 | 3.3 –3.5 | 3.10 | 85 | 91 | 41.0 | 0.0090 | 200 | 114 | 1.80 | 1.00 | 95 | 10.6 | 0.253 | .. |
| 1830C | Vitron AAA | 15 | 3.3 –3.5 | 2.85 | 85 | 91 | 41.7 | 0.0095 | 186 | 99 | 1.76 | 0.94 | 97 | 10.2 | 0.244 | .. |
| 1831A | Vitron AAA | 15 | 3.2 –3.85 | 3.6 | 85 | 92 | 38.0 | 0.0090 | 122 | 83 | 1.09 | 0.75 | 119 | 13.2 | 0.235 | 30 |
| 1831B | Vitron AAA | 15 | 3.2 –3.85 | 3.2 | 85 | 92 | 38.0 | 0.0090 | 181 | 112 | 1.63 | 1.01 | 115 | 12.8 | 0.236 | .. |
| 1831C | Vitron AAA | 15 | 3.2 –3.85 | 3.1 | 85 | 92 | 35.8 | 0.0090 | 196 | 109 | 1.77 | 0.99 | 97 | 10.8 | 0.220 | .. |
| 1831D | Vitron AAA | 15 | 3.2 –3.85 | 2.9 | 85 | 92 | 38.0 | 0.0090 | 224 | 125 | 2.02 | 1.12 | 110 | 12.2 | 0.235 | .. |
| 1832A | Vitron AAA | 15 | 3.0 –3.5 | 3.3 | 85 | 90 | 34.0 | 0.0092 | 180 | 99 | 1.66 | 0.90 | 102 | 11.1 | 0.243 | 33 |
| 1832B | Vitron AAA | 15 | 3.0 –3.5 | 3.2 | 85 | 90 | 38.7 | 0.0088 | 198 | 104 | 1.74 | 0.92 | 95 | 10.8 | 0.244 | .. |
| 1832C | Vitron AAA | 15 | 3.0 –3.5 | 3.0 | 85 | 90 | 37.0 | 0.0084 | 222 | 113 | 1.86 | 0.95 | 98 | 11.7 | 0.245 | .. |
| 1851 | Vitron AAA | 30 | 2.75–3.0 | 3.0 | 45–51 | 60 | 35.7 | 0.0090 | 223 | 160 | 2.00 | 1.44 | 102 | 11.3 | 0.221 | .. |
| 1834A | Vitron 322 | 25 | 3.0 –4.0 | 3.65 | 85 | 90 | 39.3 | 0.0095 | 157 | 88 | 1.49 | 0.84 | 106 | 11.2 | 0.230 | 33 |
| 1834B | Vitron 322 | 25 | 3.0 –4.0 | 3.5 | 85 | 90 | 37.0 | 0.0090 | 179 | 93 | 1.61 | 0.83 | 109 | 11.1 | 0.229 | .. |
| 1834C | Vitron 322 | 25 | 3.0 –4.0 | 3.2 | 85 | 90 | 38.1 | 0.0090 | 242 | 123 | 2.18 | 1.11 | 106 | 11.8 | 0.237 | .. |

[a] Determined by Bauer-McNett classifier.

below, the white water becomes cloudy, indicating that fines were leaving the paper.

In the previous publication on glass paper it was stated that the more "fines" in the paper, the weaker the paper becomes. With the addition of acid, however, it is believed that the acid works on the "fines" and turns them into a gelatinous silica which binds the fibers together, producing a paper with greater tensile strength than one containing the same proportion of "fines" but not treated with acid.

For the series 1834, a new fiber designated as 322 was used. It has the same diameter as the AAA fiber, but the fibers appear to be somewhat longer, and the glass was said by the manufacturer to be low in sodium content. At a headbox pH of 3.2 the tensile strength of the paper was 242 p.s.i., and the slope of the line is 20 p.s.i. increase in tensile strength for a decrease of 0.1 in pH. This indicates that it should be possible to attain a tensile strength of 300 p.s.i. at a pH of 2.9.

Series no. 1851 was made to see what effect the acid would have on the tensile strength of the paper using cold water (45 to 51°F.). The data in Table III show that it took twice as long to disperse the fibers at the lower temperature, with no gain in tensile strength. The data also indicate that the alkaline material is leached out of the fibers much faster at 85°F. than it is at 51°F., for at 85°F., the pH value increased 0.5 unit in 15 min., while at the lower temperature it increased only 0.25 unit in 30 min.

### *Effects of Different Concentrations of Stock at the Headbox*

Series no. 1865 was made to study the effect of different concentrations of the stock at the headbox on the tensile strength of the paper. Before making this series of runs the apron was pulled back from $^1/_2$ to $2^1/_2$ in., and the fan pump was speeded up.

Run 1865A did not look smooth on the wire. To improve this condition the jordan was closed up to a light brush, and the pH was dropped from 3.1 to 2.95 for run 1865B. (The concentration for both these runs was 0.14%.) This procedure resulted in an increase in tensile strength from 267 to 340 p.s.i. In run 1865C the concentration was increased from 0.14 to 0.20%. Table IV shows that the tensile strength dropped to 230 p.s.i. The tensile strength of run 1865C compares favorably with other runs whose conditions are similar. Therefore, the increase in tensile strength of 110 p.s.i. in 1865B can be attributed to the decrease in the concentration.

## DISCUSSION

The effect of acid in causing rapid dispersal of the fibers in the beater may be in part a static electrical phenomenon resulting from the initiation of the chemical reaction between the acid and the glass. It is known that negative electrical charges appear on the surface of the glass, and the mutual repulsion of the like-charged surfaces is probably responsible for the quick separation of the fibers as compared with their behavior when no acid is present.

As the reaction of the acid and the glass proceeds, it is known that a gelatinous, silica-rich layer is formed on the surface. It is thought that this gelatinous layer is responsible for increasing the tensile strength of the glass paper by acting as an adhesive to cement the fibers together as the paper dries. The presence of this layer is demonstrated in Fig. 3, an electron micrograph at a magnification of 6600 times. The glass fibers were taken from a run having a pH of 3.4 as it

**Table IV. Effect of Concentration of Stock at the Headbox**

Conditions for all runs: Vitron AAA fiber used; beating time 15 min.; concentration of stock in beater 0.5%; pH of stock in beater 3.2 to 3.4; temperature of stock in beater 85°F.; beater roll setting +1; temperature of stock at headbox 90°F.

| Run no. | Concentration of stock at headbox, % | Jordan setting, in. | pH at headbox | Basis weight (25 × 40—500), lb. | Thickness, in. | Tensile strength Mach. dir., p.s.i. | Tensile strength Cross dir., p.s.i. | Breaking load Mach. dir., lb./in. width | Breaking load Cross dir., lb./in. width | Air resistance Mm. | Air resistance Mm./mil | Density, g./cc. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1865A | 0.14 | 0.045 | 3.10 | 35.7 | 0.0090 | 267 | 189 | 2.40 | 1.70 | 109 | 12.1 | 0.220 |
| 1865B | 0.14 | 0 | 2.95 | 37.0 | 0.0086 | 340 | 238 | 2.91 | 2.05 | 101 | 11.7 | 0.238 |
| 1865C | 0.20 | 0 | 2.97 | 38.0 | 0.0100 | 230 | 160 | 2.30 | 1.60 | 116 | 11.6 | 0.211 |

passed onto the wire. At most of the intersections of the fibers, there are small quantities of material of apparently less density or thickness than that of the fibers. These are thought to represent the gelatinous material. They are particularly noticeable at the points where five smaller fibers project downward from the thick fiber running diagonally across the upper left quadrant of the picture, and at the intersection of several fibers in the lower left quadrant. There is also some evidence of such material between two parallel fibers running diagonally upwards toward the right center of the picture.

A pH value of approximately 4.0 seems to represent a rather critical point in this process. Above this point, it is known that hydrogen ion concentration has practically no chemical effect on glass, as it is not strong enough to leach the alkaline components from the glass. If the lines in Fig. 2 are extrapolated to pH 4.0, they cross the axis of pH 4.0 at about the tensile strength of glass paper to which no acid has been added. A more direct piece of evidence is provided by Fig. 4, an electron micrograph of glass fibers taken from a run at a pH of 3.8. There are some traces of the gelatinous material in the upper left of the picture. The irregular areas in the lower right, because of their high density, are thought to be dust, rather than gelatinous material. Nearly all the intersections, however, show clean-cut angles, with no trace of adhesive. Thus, a comparison of Figs. 3 and 4 shows that at pH 3.8 there is practically no formation of a gelatinous layer, while at pH 3.4 the occurrence of the gelatinous material is quite extensive.

The permeability of these papers to air was of particular importance since these runs were made in the course of developing a material suitable for use as a filtering medium, where the optimum permeability was between 100 and 110 mm. The need for limiting the range of permeability can be illustrated by considering the case of a filter in a gas mask or respirator for personal use. If the permeability is too high, the air

*Fig. 3. Showing the gelatinous silica-rich layer formed by the addition of acid to pH 3.4;* (×*6600*)

*Fig. 4. Showing glass fibers that were acidified to pH 3.8;* (×*6600*)

rushes through the filter so rapidly that solid particles in the air are not removed efficiently, while if the permeability is too low, the wearer must exert too much effort to inhale an adequate volume of air. The air resistance of the glass-fiber paper depends on its thickness and on the density and texture of the paper. The air resistance values are given in the tables to indicate how closely the products of the various runs met this permeability requirement.

It is of interest to consider the pH of the finished paper, as some of its possible uses might bring it in contact with metals where acidity might cause corrosion, or involve other applications where the low pH would be harmful. In a typical run with the pH at the headbox having a value of 3.0, the pH as the paper entered the first press was 4.8, and after the driers with 45% moisture in the paper its pH was 6.0. The pH of the completely dried paper was about 7.0. For several lots of finished paper, the pH has been found to lie between 7.0 and 7.5. Apparently the major part of the acid leaves the fibers with the white water, and most of the remainder reacts with the glass. It seems that the acid is only on the surface of the fibers, rather than being absorbed into the fibers, as is the case with the usual papermaking fibers, so that when the surface acid is removed by drainage of the solution or reaction with the glass, the fiber is left substantially neutral.

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# Arsenic Content of Pulp from Chemically Debarked Trees

ROBERT F. SMITH and KARSTEN MATHISEN

**Arsenic analyses have been carried out on trees which were chemically debarked, with sodium arsenite on pulp made from these trees, and on pulp to which arsenite was added at the start of the pulping process. In all cases the arsenic content of the material was well below the concentration allowable in food.**

DURING the last decade there has been an increasing interest in the use of sodium arsenite to loosen the bark on standing trees (*1–6*). Since some of the pulp made from these trees may be used in the manufacture of food wrappers or modified cellulose for food thickeners, it is important to know how much arsenic remains in the finished product. It is also important to know how much arsenic remains in the spent pulping liquors. To answer these questions a number of samples of wood pulp and liquors have been analyzed during the past 2 years. This paper reports the results of these analyses.

## METHODS AND MATERIALS

The commercial method used in chemical debarking involves the removal of a 6-in. girdle of bark from the tree and the painting of the exposed wood with 40% sodium arsenite solution (*3, 4, 6*). Samples received from cooperating paper companies were all from trees treated in this manner. In addition, trees were treated in the Huntington Forest of the College of Forestry in the Central Adirondacks and pulps were prepared from this wood. These experimental treatments were made by bathing the exposed wood of a girdle in a reservoir of sodium arsenite by means of a rubber dam placed around the girdled area.

In addition to the pulps made from treated trees, some batches were prepared from untreated wood with known amounts of arsenic added to the chips before pulping.

Wood or pulp samples were digested in hot concentrated nitric and sulphuric acid. Samples of pulping liquors were evaporated to low volume with added nitric acid before fuming with sulphuric acid. Arsenic was distilled from the digestion mixtures as the trichloride and the concentration determined colorimetrically by the molybdenum blue method (*7, 8*).

When the amount of arsenic in the sample was expected to be less than 10 μg., the distillates were evaporated to dryness with nitric acid and the volume of molybdate solution kept to a minimum. When handled in this way, a difference in color intensity corresponding to 0.2 μg. of arsenic could easily be determined on the colorimeter. This means that in a 10-gram sample 0.02 p.p.m. of arsenic could be detected. However, addition of small known amounts of arsenic to pulp and liquor samples before digestion revealed that there might be a loss of as much as 0.5 μg. of arsenic during the digestion and distillation (Table I).

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**Table I. Recovery of Arsenic Added Before Analysis**

| *Sample* | *Total As found, μg.* | *Added As recovered, μg.* |
|---|---|---|
| 10 ml. acid from accumulator pump (no As added) | $0.2_5$<br>$0.0_5$ | |
| Same—with 2 mg. As added | 1.65 | 1.5 |
| 7 grams unbleached sulphite pulp (no As added) | 0.2 | |
| Same—with 2 mg. As added | 2.2 | 2.0 |
| 8 grams bleached sulphite pulp (no As added) | 0.0 | |
| Same—with 2 mg. As added | 1.7<br>1.9 | 1.8 |

## RESULTS

The slight degree of penetration of arsenic into the wood of spruce is illustrated by the figures in Table II. The samples were taken 1 ft. above or below a reservoir of arsenite solution which was in contact with the unscored sapwood. The trees were treated in the Huntington Forest in late April or early May and sampled within 2 weeks while the reservoirs still contained arsenite solution. The amount of arsenic that could soak into the tree under these conditions is much greater than is available in the thin film of solution applied by paint brush in the commercial method.

Table III shows the arsenic content of some trees which were treated by the commercial method (arsenic solution applied by paint brush on an unscored girdle). Samples were taken 4 to 10 months after treatment.

One of the companies sponsoring the chemical debarking project at the College of Forestry sent in a large number of samples from both chemically peeled wood (indicated by the letters "S.T."—for special test) and untreated wood. Samples consisted of batches of chipped wood, pulp in various stages of processing, and pulping liquors. The analyses of some of these are shown in Table IV. Since there is little arsenic in any of these samples, a loss of the magnitude shown in Table I might seem relatively large, so figures in parentheses show the results corrected for a loss of 0.5 μg. of arsenic in the analysis.

**Table II. Penetration of Arsenic in Trees Treated by Reservoir Technique**

| *Sample position* | *Tree No. 827* | | *Tree No. 819,* |
|---|---|---|---|
| | *Above girdle, p.p.m. As* | *Below girdle, p.p.m. As* | *above girdle, p.p.m. As* |
| Inner bark | 4080 | 26 | 7,750 |
| Wood, 0–0.5 mm. from cambium | 7625 | 9 | . . . . |
| | | | 13,800 |
| 0.5–1.0 mm. from cambium | 2042 | | |
| 2–3 mm. from cambium | 30 | | . . . . |
| 2–5 mm. from cambium | . . . | | 9.6 |

**Table III. Distribution of Arsenic in Wood of Trees Treated by Commercial Method**

| Sample position | Arsenic content in p.p.m. (mg. As/kg. dry wt.) Yellow birch | Yellow birch | Hemlock | Red spruce | Balsam fir |
|---|---|---|---|---|---|
| Wood surface | | | | | |
| Below girdle | 2.3 | 7.9 | ... | 6.6 | ... |
| At girdle | 187 | ... | ... | 6.0 | ... |
| 4 ft. above girdle | 10.3 | 7.5 | 4 | 6.9 | 1.8 |
| 16 ft. above girdle | ... | 12 | 5.6 | 6.9 | ... |
| 1–3 mm. into the wood, 18 in. above girdle | ... | 0 | 2.7 | 2.0 | ... |
| 1 cm. into wood 4 ft. above girdle | 0.93 | ... | ... | 2.0 | ... |

Not one of these samples contains more than 0.5 mg. of arsenic per kilogram of dry material (or per liter of the liquids) even allowing for the maximum observed loss during analysis of 0.5 μg. arsenic per sample.

Another sponsoring company has submitted samples of hardwood chips and sulphite pulp prepared from chemically peeled wood. Table V gives the results of analyses of this material, compared with analyses of untreated trees.

We have no explanation for the increase in arsenic in the bleached pulp except for the possibility that some arsenic was present in the bleaching chemicals.

A third sponsoring company submitted samples of bleached and unbleached pulp from chemically debarked trees. Both samples gave negative tests for arsenic which, with the method used, means that they had less than 0.2 p.p.m. of arsenic.

Tables VI and VII give the arsenic contents of experimental batches of pulp prepared in the pulp and paper laboratory of the College of Forestry. Trees no. 323 (spruce) and no. 365 (birch) were treated in the Huntington Forest by the reservoir method mentioned above. The butt bolts of trees nos. 323 and 365 were used. The control batches shown in Table VI were made from chips of untreated trees with known amounts of arsenic added to the pulping liquors.

**Table IV. Arsenic Content at Various Stages of Pulping Chemically Debarked and Untreated Wood**

| | Arsenic content, p.p.m.[a] Using chemically peeled wood | Using untreated wood |
|---|---|---|
| Softwood chips chemically peeled trees (three batches) | 0.30 (0.40)<br>0.35 (0.45)<br>0.30 (0.45) | ...<br>...<br>... |
| Groundwood pulp | 0.30 (0.50) | 0.30 (0.50) |
| Digester acid from sampling line, 3rd S.T.,[b] 4th hr. | 0.07 (0.12) | ... |
| Digester acid, 1st cook following S.T., at blow | ... | 0.17 (0.22) |
| Digester acid 2nd cook following S.T., at blow | ... | 0.11 (0 16) |
| Acid from accumulator pump during S.T. | 0.02 (0.07) | ... |
| Acid from accumulator pump before S.T. | ... | 0.11 (0.16) |
| Jonsson screen rejects | 0.35 (0.44) | 0.06 (0.17) |
| Jonsson screen rejects | 0.08 (0.17) | ... |
| Chlorinated stock after 1st stage thickener | 0.10 (0.19) | 0.03 (0.25) |
| Caustic extracted stock | 0.25 (0.50) | 0.10 (0.20) |
| Caustic extracted stock, 1 hr. after S.T. | ... | 0.03 (0.45) |
| Unbleached sulphite pulp | 0.00 (0.1) | 0.00 (0.1) |
| Unbleached sulphite pulp, after S.T. | ... | 0.00 (0.1) |

[a] The figures in parentheses are the corrected figures for a *possible* loss of 0.5 μg. arsenic in the analysis.
[b] S.T., meaning special test, indicates runs on chemically debarked wood.

**Table V. Arsenic Content at Various Stages of Pulping Chemically Debarked and Untreated Hardwoods**

| Sample | Arsenic content in p.p.m. From chemically debarked trees | From untreated trees |
|---|---|---|
| Hardwood chips | 0.90[a] | 0.75,[b] 0.54[c] |
| Spent liquor | 0.14 | ... |
| Unscreened pulp from 3rd stage black liquor washer | 0.33 | ... |
| Brown stock pulp, screened | 0.20 | 0.20 |
| Bleached pulp from white stock washer | 1.10 | 0.20 |

[a] Mixed hardwoods.
[b] Yellow birch from Adirondacks.
[c] Yellow birch from Cortland County, N. Y.

## DISCUSSION AND CONCLUSION

The legal tolerance of lead arsenate spray residues on apples and pears was set in 1940 at 0.025 grain $As_2O_3$ per pound or 2.7 p.p.m. of arsenic (*9*). We have analyzed some foods for arsenic and found 0.6 p.p.m. in a California burgundy, 0.8 p.p.m. in a Canadian cider, and 4.5 p.p.m. in fresh shrimp. It is apparent from the results tabulated above that pulp and paper from chemically debarked trees contain no more and usually less arsenic than common foods and are safely within the tolerance limits allowed.

The arsenic content of chips from treated trees was found to be less than 1.0 p.p.m. in all samples tested (Tables IV and V). At first glance, this seems surprisingly low. Taking 200 kg. as the approximate weight of the merchantable wood in a red spruce of $10^1/_2$ in. dbh., 1 p.p.m. of arsenic would be 0.2 gram per debarked log. Felix Czabator, forester of the chemical debarking project, has estimated the average amount of arsenic used per tree on the basis of the number of gallons of Atlas A used and the number of trees treated by companies cooperating in the chemical debarking experiments. For a $10^1/_2$-in. spruce about 10 grams of arsenic are used. Apparently less than 2% of it turns up in the merchantable wood. A number of suggestions may be made as to where the rest of it went: (1) the estimate of arsenic consumption per tree includes spillage—which may be considerable, (2) quite a lot of arsenic goes up into leaves, buds, twigs, and small branches and (in hardwoods) down into the roots, (3) of the residual arsenic in the bole of the tree probably 90% is in the bark. The concentration of less than 1.0 p.p.m. arsenic in mixed chips is consistent with the concentrations found in the surface wood as reported in Table III.

The experimental batches of pulp for which the analyses are shown in Tables VI and VII were prepared from butt bolts of reservoir-treated trees. It is doubtful if any bolts of commercially treated wood will ever

**Table VI. Arsenic Contents of Experimental Pulps from Reservoir Treated Spruce**

| | Arsenic concentration in p.p.m. Sulphite pulp | | Sulphate pulp | |
|---|---|---|---|---|
| | Control (As added[a]) | No. 323 | Control (As added[b]) | No. 323 |
| Spent liquor | 9.0 | 2.1 | 5.6 | 3.3 |
| Unscreened pulp | 1.1 | 0.94 | ... | 1.5 |
| Screened pulp | ... | 0.3 | 3.0 | 1.45 |
| Pulp after 3rd bleach | ... | 0.2 | ... | 0.2 |

[a] 0.1 mg. arsenic per gram of dry wood added.
[b] 0.032 mg. arsenic per gram of dry wood added.

**Table VII. Arsenic Contents of Experimental Pulps from Reservoir Treated Yellow Birch**

| *Sample (yellow birch no. 365)* | *Arsenic concentration in p.p.m.* Sulphate pulp | Soda pulp |
|---|---|---|
| Black liquor | 4.3 | 4.7 |
| Screened pulp | 2.8 | 2.0 |
| After beating | 1.8 | 1.8 |
| After bleach, 2nd stage | 0.2 | 0.2 |

contain as much arsenic as these. But even in this extreme case the arsenic remaining in the finished pulp was negligible.

Analyses of pulps and liquors from commercial operations (Tables IV and V) show that the arsenic content of all these materials is negligible.

These analyses have been reported at some length because of the importance of establishing with as much certainty as possible the answers to the questions stated in the introduction—whether or not there is danger of getting harmful amounts of arsenic in products from chemically debarked wood. The results of this work indicate that there is no such danger.

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# The Accelerated Biological Oxidation of Kraft Mill Effluents


FRED W. BISHOP



**As a result of the laboratory and pilot plant work of the National Council for Stream Improvement and with their guidance, Southland Paper Mills has installed a full mill scale treatment plant employing the aeration process for accelerated B.O.D. reduction of kraft mill wastes. This reduction occurs at a high rate when the waste is exposed to sufficient quantities of diffused air in the presence of acclimated bacteria with nitrogen being fed to sustain the activity of the seed. In this instance, the waste originates from the decker seal pit with a B.O.D. range of 200 to 500 p.p.m. With the incoming strength sustained at 200 p.p.m. an operating reduction of 85% can be expected, and with a strength of 500, a reduction of 60% is more general. A properly designed system can be operated with a minimum of attention and maintenance. Further advantages can be derived when this process is employed in conjunction with impounding or detention basins where B.O.D. reduction occurs naturally at a much slower rate. When the clarified effluent from the accelerated process is added to larger volumes of raw waste, the oxidation rate of the mixture is greatly stimulated, reducing the detention period requirement by as much as 75%. It is the opinion of authoritative observers that this installation has proved the feasibility of the accelerated process within the bounds of economic justification.**


The bacteriological decomposition of kraft mill wastes will occur quite naturally if sufficient time is allowed for this reaction to proceed to completion. Relating to the waste disposal problems of kraft mills, the most pressing need of the industry was to have available a process which could cause these time-consuming processes to occur at greatly accelerated rates. The National Council for Stream Improvement assigned this problem to their Southern Regional Laboratory at Louisiana State University under the direction of William A. Moggio. The "aeration process" for the accelerated treatment of kraft mill wastes was conceived and developed from experimentation conducted by this group. Recognizing that this process might hold great promise, member mills of the southern region advanced money on the pro rata basis for the building of a pilot plant to be located at a mill and operated by the personnel of the National Council.


Fred W. Bishop, Chief Chemist, Southland Paper Mills, Inc., Lufkin, Tex.


### THE PILOT PLANT

The pilot plant substantiated the laboratory findings and was instrumental in establishing requirements, ranges of operation, retention times, capacities, operating costs, and capital investment requirements for large scale installations. Although related to the activated sludge processes, the loading efficiency with the necessary modifications was about three times that designed for treating sanitary sewage. This work indicated that further advantages can be derived when the process is employed in conjunction with impounding or detention basins where B.O.D. reduction occurs naturally at a much slower rate. When the clarified effluent from the accelerated process is added to larger volumes of raw waste, the oxidation rate of the mixture is greatly stimulated, reducing the detention period requirement by as much as 75%.

### THE PROCESS

Basically, the process is one of perpetuating the

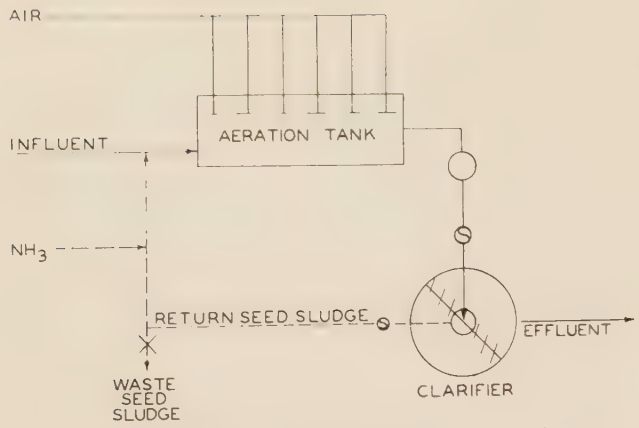


*Fig. 1. The aeration process (schematic)*

species of acclimated bacteria by supplying the necessary requirements of food, nitrogen, and air and by generally maintaining these healthful environmental conditions. The food originates from the organic matter in the waste, the nitrogen is supplied from ammonia, and oxygen is supplied by diffusing low pressure air through the total mixture for several hours. Under these conditions the microorganisms devour the organic compounds and derive their energy by the oxidation of these, thus reducing the amount of organics and correspondingly the B.O.D. of the effluent. Following the reactions in an aerator, the total effluent is clarified for the collection, concentration, and recirculation of seed sludge for admixing with new waste to aerator. With ravenous appetites sustained, the bacteria continuously reduce the B.O.D. of incoming wastes.

## THE SOUTHLAND INSTALLATION

For many years Southland Paper Mills has been making very effective use of the ponding operation whereby with throughput detention periods up to 20 days the over-all B.O.D. of total mill effluents has been reduced as much as 80%. As the decker seal pit effluent from the kraft mill was the largest single weighted contributor of B.O.D., the accelerated process was of great interest to this operation. This was particularly true since considerable idle equipment was already in existence which might be employed in the process with small conversion cost. Included in this was a concrete tank 10 by 11 by 270 ft. long and an up-flow type clarifier with a volume of 120,000 gal. With the purchase of a blower and with some minor changes in the clarifier to convert it partially to radial design, and with some extensive piping, the process was put into operation at a minimum cost. The blower is driven by a 75-hp. motor at 3600 r.p.m. delivering 1500 c.f.m. at 6 p.s.i.

A properly designed system can be operated with a minimum of attention and maintenance. The influent to the system can be controlled manually with an indicating flow meter in the line. The transfer station from the aerator to the clarifier can be operated with automatic level control equipment actuating a valve on the pump discharge. The recirculated sludge volume is normally set at a constant rate and generally can be left intact. With this system the influent flow can be changed or shut off completely with one manual valve and automatic flow compensations will be made with the sludge continuing to be recirculated, aerated, and activated (see Fig. 1).

The normal flow rate of waste to be treated never exceeds 600 g.p.m. and the B.O.D. range will vary from 200 to 500. In operation the B.O.D. level stays relatively constant for a period of 2 or 3 weeks with changes occurring gradually to move it from one level to another prior to each period of sustained strength. At low levels of B.O.D., the percentage reduction is often as much as 85%, but with incoming B.O.D. of 500, a reduction of 60% is more general.

## OPERATION

The system was seeded initially with 10,000 gal. of sanitary sewage sludge and with excess air the flow of raw waste was started slowly and increased gradually. In the operation of any activated sludge process there is always considerable concern about losing the activity of the sludge or of actually killing the bacteria. Throughout this operation on mill scale there have been many occasions when the organisms have been subjected to rather drastic surges of waste concentration and, generally, they will show some loss in effectiveness but recovery will occur in a very few days. Due to bearing failure on the blower, there have been occasions when the sludge was without air for periods of 36 hr. During a period of 3 weeks when the recovery boiler was being rebricked, seed organisms were kept alive by feeding black strap molasses to the system in the presence of the other activating requirements. This source of food was added at a rate of approximately 25% of the normal food which would be derived from waste in operation. After resuming pulp mill operation, the waste load was gradually increased to maximum flow over a period of about 5 days. It appears that once the species are established and acclimated they become very hardy as long as the operation is conducted to avoid extreme conditions.

From the aerator the seed sludge is a very rapid floccing and settling material which lends itself to sedimentation in a radial design clarifier. Due to the very large particle size of the floc, it naturally entrains large quantities of water and this does not allow much compaction in the sludge bed. In general, the under flow from a normally equipped clarifier will contain no more than 6000 p.p.m. of sludge solids. In present operation it is necessary to recirculate 400 g.p.m. of underflow sludge to the aerator to maintain a concentration of approximately 2000 parts upon admixing with the raw waste. A new clarifier is in the process of being installed which is radial in design, and the agitator arms are equipped with pickets which will facilitate dewatering of the floc particle and compaction in the bed for higher solids discharge. With solids increased and return flow reduced proportionately, the retention and contact time with new waste in the aerator will be increased.

In the aerator there has been some indication that sludge concentrations in excess of 2500 p.p.m. may actually contribute some additional B.O.D. load to the system. With this in mind, it is often necessary to bleed off some sludge volume to keep the required concentrations at optimum levels. Although the minimum air requirement has been established at 1 c.f.m. per 1 g.p.m. of new waste, this particular system has always

been operated at a figure of nearer 2 c.f.m. When more air than this is added, there have been some occasions when a trace of dissolved oxygen was present at the aerator discharge. It has been advised that the system be operated without any dissolved oxygen at this point, and accordingly the air flows were reduced to such a rate that this did not recur.

The foaming condition at the aerator is serious and has presented a very big problem. As the aeration tank is directly adjacent to a pulpwood conveyor, it was necessary to build a 4-ft. baffle the full length of this tank in order to divert the foam to the ground on the other side. Experimentation has indicated that this foam can be knocked down at the discharge side with the use of fan-type sprays. These cut and disintegrate the foam at its point of overflow and allow it all to be retained on the surface of the aerator. The water source to these sprays in final application will be derived from the aerator discharge and this will constitute a low volume recirculated flow. In present operation considerable quantities of sludge are lost in the overflowing foam, and this acts as an automatic sludge bleeding device. With the foam showers working properly, it is anticipated that excess sludge must be bled from the system at frequent intervals.


### SUMMARY

The final effluent from the overflow of the clarifier is reduced in B.O.D. load by 60 to 85% and is relatively stable to any immediate oxygen demand. With the accelerated process in operation on kraft mill wastes and biological detention ponds handling total mill effluents, the mill wastes are discharged to the stream at 30 p.p.m. of B.O.D., whereas from ponding operation alone, they were formerly entering at 60. Due to the large volumes of waste to be treated in a normal kraft mill, the capital expenditure for the installation of such a process would be quite high, and the final use of it would be governed by the intensity of the problem.



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# The Beater Addition of Phenolic-Rubber Combination, Part I


R. KRAJEWSKI and V. STANNETT



**A number of phenolic resin-synthetic rubber blends have been investigated as beater additives. The effect of these blends on the wet and dry mechanical properties of paper is reported and compared with the properties imparted by either component used alone. In general, the use of blends gave mechanical properties considerably in excess of the separate components. The effects of various conditions of heat cure on the treated papers have been studied and the optimum cure conditions determined. Other variables such as the freeness of the pulp used and the effect of the resin-rubber blends on the stock properties are also discussed.**


The blending of synthetic rubbers with phenol-formaldehyde resins has become an important development in the plastics and rubber industries within the last few years. Phenolic resins are hard, strong, but relatively brittle materials, whereas rubbers are soft, extensible, and extremely tough. It was natural therefore that a combination of the two might produce polymer blends of considerable value. The blending of such dissimilar polymers initially proved a failure owing to their lack of mutual compatibility. However, compatible blends are now possible using specially modified phenolics and more polar rubbers, in particular, the butadiene-acrylonitrile copolymers (*1*). Such blends have become of great value in the fields of phenolic modified rubbers and rubber modified phenolic for molding and adhesive applications.

There is little information, however, on the beater addition of such combinations to paper. This article describes some of the properties produced by such an addition.


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### MATERIALS AND METHODS

Western hemlock unbleached sulphite and southern pine unbleached kraft were used for the investigation. The resins and rubbers used together with their properties are listed in Table I. Percentage additions were calculated on the oven-dry weight of pulp.

The phenolic-rubber mixtures were added as a combined 1% solids content emulsion to the beaten pulp at 2% consistency with stirring. After 5 min. of stirring the resins were precipitated by adding 4% alum solution in a thin stream until a pH of 4.5 was reached. A freeness test (Canadian Standard) was then made and handsheets formed using a British Standard circular sheet mold with a white water recirculatory system. The handsheets were cured at the specified

**Table I. Resins and Rubbers Used**

| *Material* | *Form supplied* | *Manufacturers' designation* | *Solids content, %* | *Remarks* |
|---|---|---|---|---|
| Phenol-formaldehyde resin | Emulsion | Durez 14136 | 40.1 | |
| Phenol-formaldehyde resin | Water solution | Durez 14798 | 61.5 | Emulsifies on mixing with latex and diluting |
| Butadiene-acrylonitrile copolymer rubber | Latex | Hycar 1561 | 39.3 | Higher acrylonitrile content |
| Butadiene-acrylonitrile copolymer rubber | Latex | Hycar 1562 | 38.3 | Lower acrylonitrile content |

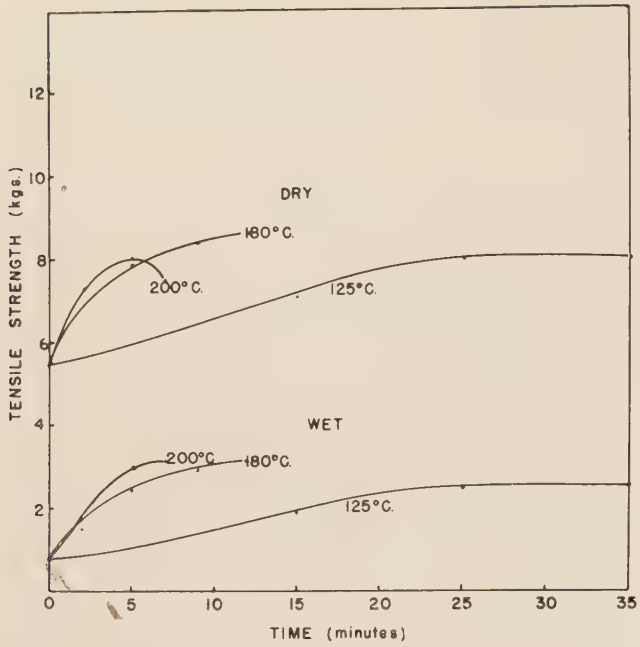


*Fig. 1. Effect of curing conditions on wet and dry tensile strength of unbleached sulphite treated with 5% Durez 14798 + 5% Hycar 1561*

time and temperature and then conditioned for 24 hr. at 73°F. and 50% R.H. before testing.

## EFFECT OF HEAT CURING CONDITIONS

Each of the resins and latices used required heat treatment to develop their maximum mechanical properties. This necessity has been previously reported by Oliner and O'Neil (*2*). The following reasons may be put forward in explanation of this effect: (1) to reduce the moisture content of the system thus bringing the resin particles closer in contact with the fibers, (2) to reach the softening point of the resins enabling them to flux into a more continuous state among the fibers, (3) to promote crosslinking of the polymers, and (4) to promote reaction between the resin and the elastomer in the case where combinations are used.

The effect of heat curing under different conditions is shown graphically in Figs. 1 to 4. It can be seen that the dry burst strength reaches approximately its maximum at about 120°C. although longer heating is required at lower temperatures. Treatment at a temperature of about 180°C. is required to bring the tensile and wet strengths to their maximum values. For the comparative studies reported in this paper cure was standardized at 9 min. at 180°C. This is, of course, a very drastic treatment from the papermaker's point of view and current research is aimed at reducing the curing conditions to those of the papermaking machine. Initial results of this work which will be the subject of a later communication show this may be possible.

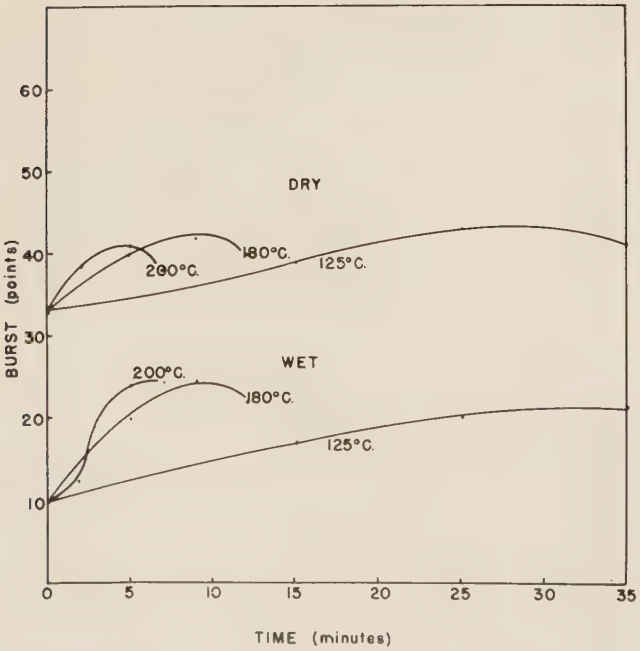


*Fig. 2. Effect of curing conditions on wet and dry burst strength of unbleached sulphite treated with 5% Durez 14798 + 5% Hycar 1561*

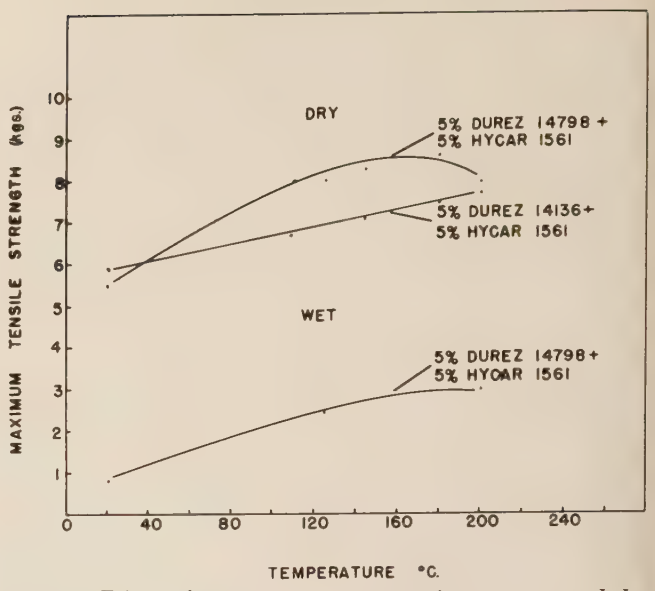


*Fig. 3. Effect of temperature on maximum wet and dry tensile strengths (sulphite)*

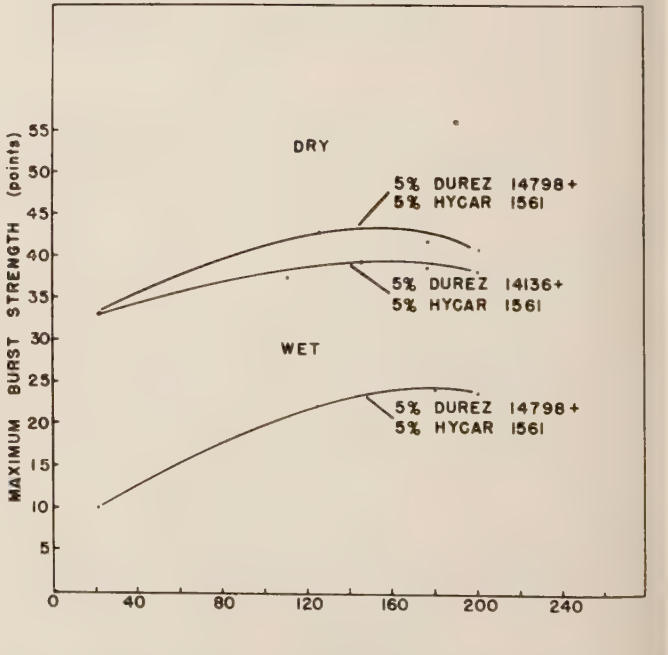


*Fig. 4. Effect of temperature on maximum wet and dry burst strengths (sulphite)*

Table II. Effect of Rubber-Phenolic Ratio on Various Properties

| Parts per 100 dry (unbleached sulphite) pulp: Rubber Hycar OR-15 1561 | Phenolic Durez 14798 | % Stretch | Tear factor | Folding endurance no. | Abrasion loss, mg./1000 rev. | Cobb size (60 sec.), g./sq. m. |
|---|---|---|---|---|---|---|
| 10 | 0 | 3.1 | 0.62 | 284 | 18 | 13.7 |
| 7.5 | 2.5 | 3.0 | 0.66 | 448 | 15 | 12.6 |
| 5.0 | 5.0 | 3.0 | 0.70 | 303 | 10 | 13.6 |
| 2.5 | 7.5 | 2.9 | 0.58 | 292 | 16 | 15.4 |
| 0 | 10 | 2.7 | 0.64 | 395 | 20 | 15.8 |
| Untreated | | 2.9 | 0.80 | 84 | 40 | 56.1 |

## EFFECT OF RESIN-RUBBER RATIO

A standard addition of 10% total resin content was made to the pulp and the ratio of phenolic resin to rubber varied over the complete range. The results of these studies are shown in Figs. 5 and 6. It can be seen that a pronounced improvement in dry tensile and burst strengths is obtained by blending the phenolic and rubber. The wet strengths are shown in Fig. 7 and are not so conclusive although one combination shows definite improvement in wet strength. Other properties including Cobb size, abrasion, fold, stretch, and tear strengths have been measured and in general the combination of resin and rubber shows properties lying roughly between those of the single resins used alone. Results of the two resins used alone and of the 50:50 blend are shown in Table II. Maximum reinforcement appears to occur in the tensile and burst strengths when about 50% of each component is present. The greatest improvements were found when the single-stage, water-soluble resin Durez 14798 was used although whether this is due to its single stage or water-soluble nature was not determined. Two-stage resins are difficult to evaluate in the sense that it is hard to add the necessary extra hexamine without its being carried away in the white water.

Although the effect of the acrylonitrile content of the butadiene-acrylonitrile rubber was not specifically

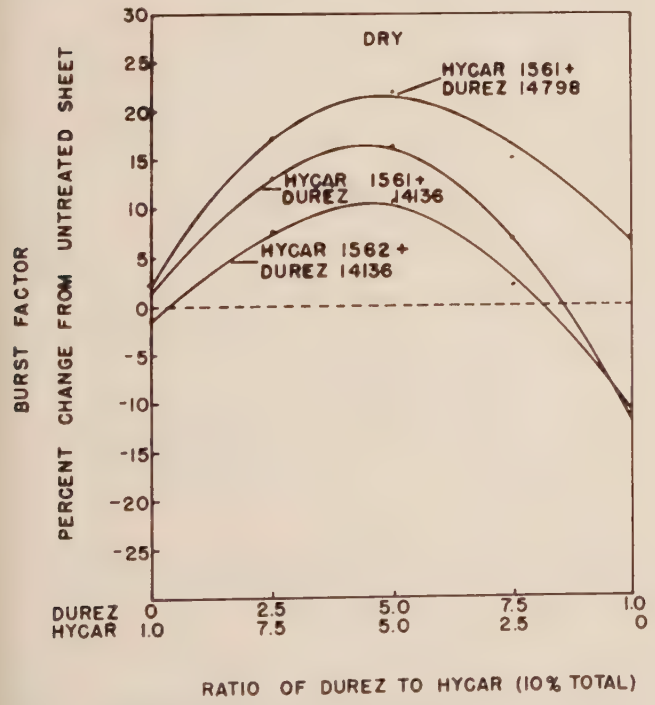


*Fig. 5. Effect of rubber-phenolic ratio on dry burst strength (sulphite)*

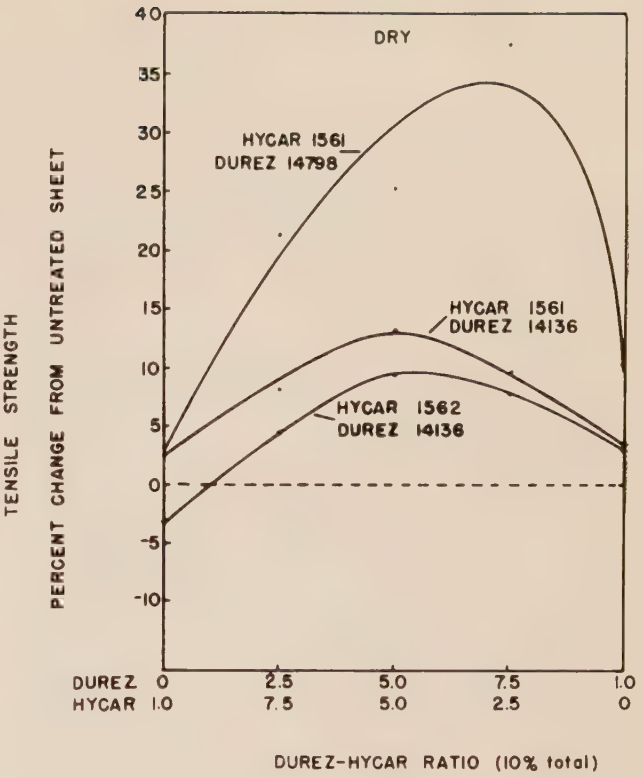


*Fig. 6. Effect of rubber-phenolic ratio on dry tensile strength (sulphite)*

studied it was found that Hycar 1561 which contains the higher acrylonitrile content gave consistently higher results than Hycar 1562. This is in line with the results of Yost and Aiken (*3*) and other workers (*1*).

## EFFECT OF AMOUNT OF LATEX BLEND

Varying amounts of the two best 50:50 blends were

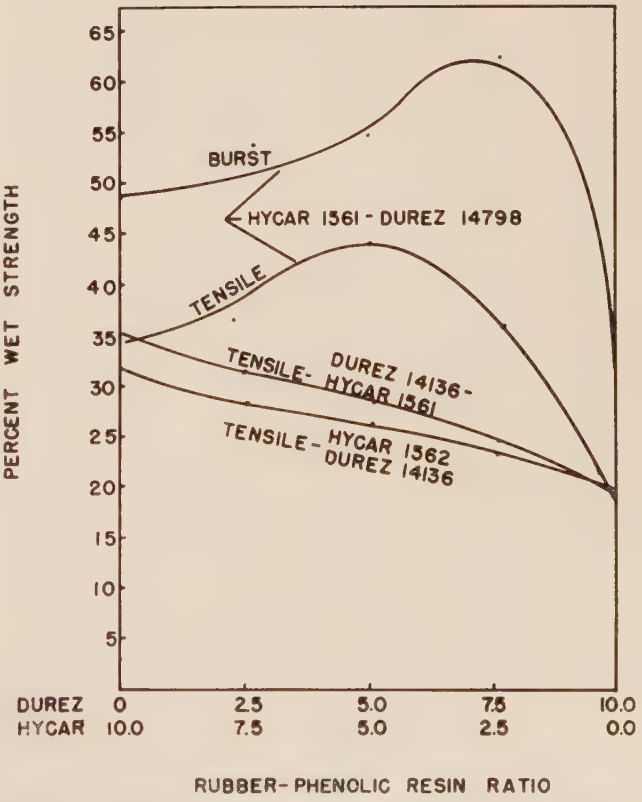
BURST
HYCAR 1561 - DUREZ 14798
TENSILE
TENSILE - DUREZ 14136 - HYCAR 1561
TENSILE - HYCAR 1562 DUREZ 14136
PERCENT WET STRENGTH
DUREZ 0 2.5 5.0 7.5 10.0
HYCAR 10.0 7.5 5.0 2.5 0.0
RUBBER-PHENOLIC RESIN RATIO

*Fig. 7. Effect of rubber-phenolic ratio on wet strength (sulphite)*

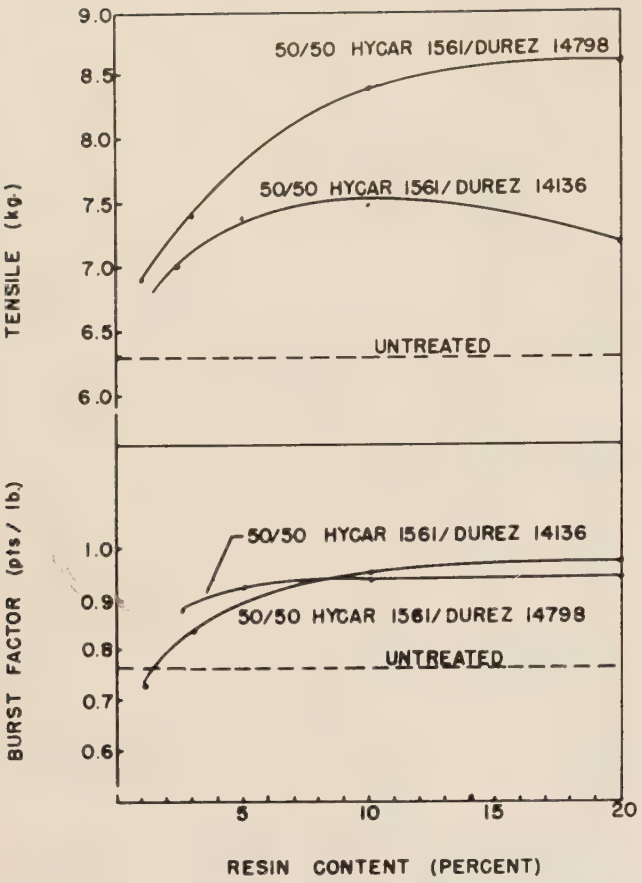


*Fig. 8. Effect of amount of latex blend on dry strength (sulphite)*

added to pulp in the manner described above and the properties of the paper determined. The results are plotted in Figs. 8 and 9. The wet and dry strength properties, in general, tend to level off at a certain resin

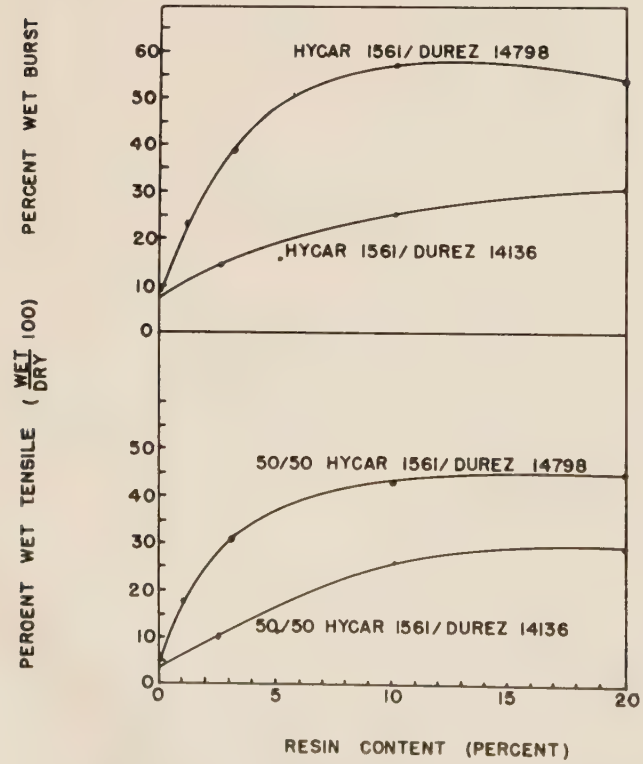
HYCAR 1561/ DUREZ 14798
HYCAR 1561/ DUREZ 14136
50/50 HYCAR 1561/ DUREZ 14798
50/50 HYCAR 1561/ DUREZ 14136
PERCENT WET BURST
PERCENT WET TENSILE (WET/DRY 100)
RESIN CONTENT (PERCENT)

*Fig. 9. Effect of amount of latex blend on wet strength (sulphite)*

**Table III. Effect of Rubber-Phenolic Blend on Stock Properties**

| *Hycar 1561* | *Durez 14798* | *Durez 14136* | *Drainage time, sec.* | *Canadian Standard freeness, ml.* |
|---|---|---|---|---|
| 10 | 0 | 0 | 11.0 | 310 |
| 7.5 | 2.5 | 0 | 16.0 | 261 |
| 5.0 | 5.0 | 0 | 15.0 | 240 |
| 2.5 | 7.5 | 0 | 14.0 | 260 |
| 0 | 10.0 | 0 | 13.0 | 265 |
| 7.5 | 0 | 2.5 | 16.0 | 220 |
| 5.0 | 0 | 5.0 | 17.0 | 210 |
| 2.5 | 0 | 7.5 | 16.0 | 210 |
| 0 | 0 | 10.0 | 15.0 | 260 |
| 0.5 | 0.5 | 0 | 14.0 | .. |
| 1.5 | 1.5 | 0 | 14.0 | .. |
| 2.5 | 2.5 | 0 | .. | .. |
| 5.0 | 5.0 | 0 | 15.0 | .. |
| 10.0 | 10.0 | 0 | 17.0 | .. |
| 0 | 0 | 0 | 14.0 | 280 |

content. This is similar to the results of many other workers (see, for example, (*2*) and (*3*)). The results in every case were above those of the untreated paper.

## EFFECT OF BEATING

Ten per cent of the best resin-rubber blend was added to unbleached sulphite and kraft pulps which had been beaten to various Canadian Standard freenesses down to 125 and 153 ml., respectively. Handsheets were prepared and cured as before. The wet and dry strength properties are plotted against freeness in Figs.

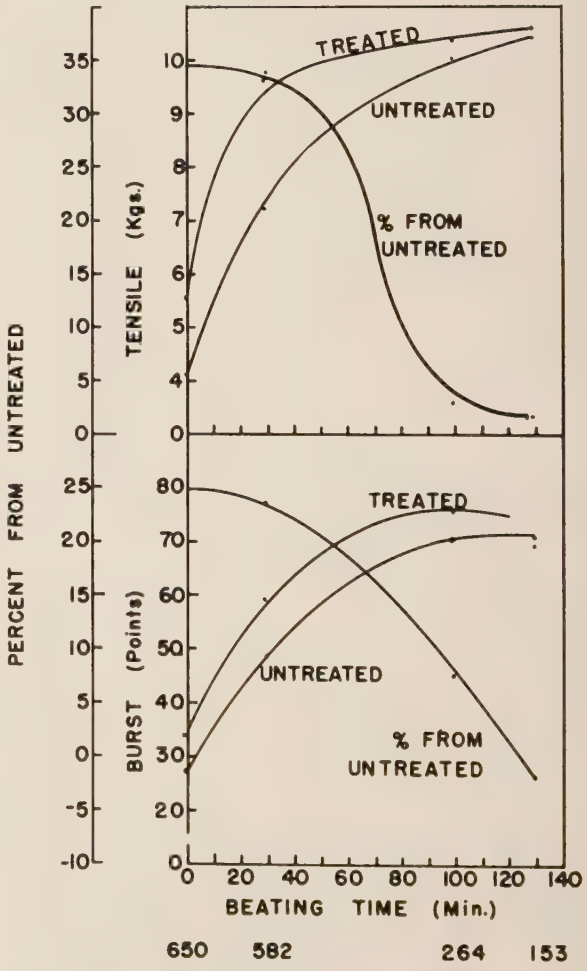


*Fig. 10. Effect of beating on dry strength (kraft pulp)*

10 and 11. It can be seen that the lightly beaten pulps benefited most from the addition of resin. However, the actual strength (as opposed to per cent improvement over untreated) rose with increased beating to an approximately stationary value. The results of beating may be described here as consequence of several competing factors namely: (1) the formation of greater surface area by fibrillation increasing the number of fiber-fiber bonds and also possible resin-fiber bonds, (2) the separation of fibrillae by resin particles causing a reduction in fiber-to-fiber bonding. Thus with lightly beaten pulps the number of fiber-fiber bonds is comparatively small and the addition of resin grossly improves the dry strength. Continued beating produces a gradual increase in the number of water-resistant resin bonds as evidenced by an increase in wet-strength values. The rate of this increase is slow while the number of fiber-fiber bonds increases rapidly with beating. At high degrees of beating the resin-fiber bonds are formed at the expense of fiber-fiber bonds possibly due to separation of fibrillae by resin.

## EFFECT OF RESIN-RUBBER BLENDS ON STOCK PROPERTIES

One of the serious disadvantages of addition of

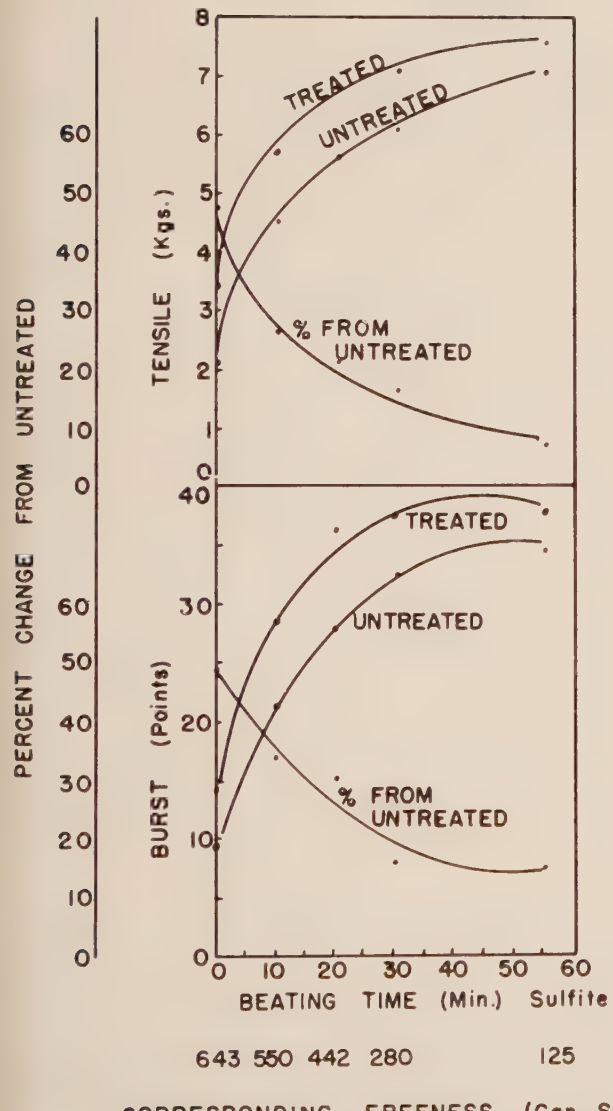


*Fig. 11. Effect of beating on dry strength (sulphite pulp)*

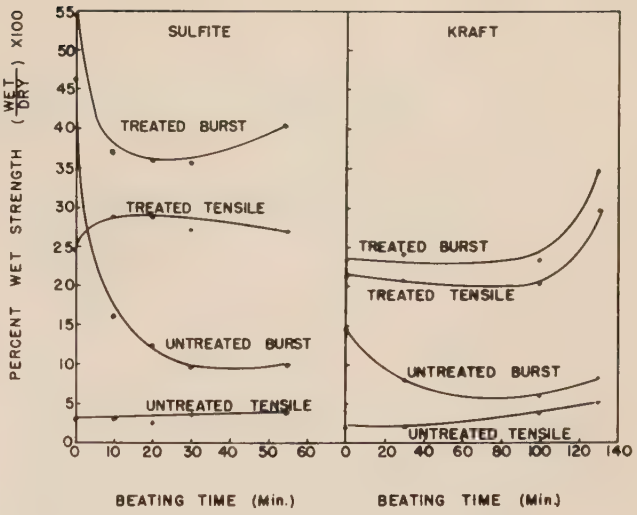


*Fig. 12. Effect of beating on wet strength*

resinous materials to pulp is slow drainage on the machine. It was necessary, therefore, to make an analysis of the effects of the resin-rubber combinations on drainage time and freeness. Typical results are shown in Table III. It can be seen that the resin-rubber combinations are slower draining than either used alone, but that no great increase in drainage time was found even for the slowest case. In the same table the effect of increasing resin content is shown, the combinations are all slower draining than the straight rubber added at the same concentration.

## SUMMARY


The following conclusions may be drawn from the work described above: (1) The synthetic rubber-phenolic combinations require heat treatment to develop maximum wet and dry strength properties. (2) The dry strength of treated papers was greatest when a combination of phenolic resin and synthetic rubber was used, i.e., the blend gave considerably better properties than either polymer used alone. (3) The wet-strength properties showed the blend to be superior only when a single-stage phenolic was used. With the two-stage resins wet properties intermediate between those of the single polymers were found. The greatest reinforcing action in both dry and wet properties was shown by the single-stage water-soluble phenolic resin. (4) Both wet and dry strength properties increased with increasing resin content up to a certain resin content and then tended to level off in the conventional manner. (5) The effect of beating was to increase the dry burst and tensile strengths of the treated and untreated papers. However, the percentage change between the treated and untreated papers decreased strongly with increased beating. The wet-strength properties increased only slightly with greater beating time. (6) The addition of resin-rubber combinations to pulp resulted in a decreased freeness and increased drainage time. However, these amounted even in the worst case to less than a 25% change.


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Received June 21, 1954.

# Studies in Continuous Alkaline Pulping

## *IV. The Effect of Continuous Methods on Liquor Evaporation and Salt Cake Losses*

W. J. NOLAN and G. R. KULKARNI

**Continuous methods of pulping are compared to industrial batch methods from the standpoint of chemical usage and recovery. A series of equations is developed by which the evaporation loads may be calculated for each system in combination with different washing systems. Continuous pulping at high concentration (80 grams per liter) for dissolving grades requires 32% more evaporation, uses 85% more chemical, and requires an additional stage of washing over the requirements of batch pulping, but does not require the prehydrolysis usually used in the production of dissolving grades. Continuous pulping at low concentration (20 grams per liter) for papermaking grades does not increase either evaporation load or chemical usage over the requirements of batch pulping. Chemical losses in the washed pulp are higher but do not warrant the use of an additional washing stage.**

THE process of continuous alkaline pulping, involving the preshredding of chips and the cooking of these chips at sustained concentration and constant temperature, has been under investigation for the past 4 years. Several papers (*1*, *2*, *3*) have been published on the subject, all dealing with the results of small scale pulping experiments. Numerous discussions have taken place in which the question of cost of this process relative to present batch operation has predominated. The use of concentrations as high as 80 grams $Na_2O$ per liter and the sustaining of such concentrations throughout the cook suggest that very large quantities of chemical may be involved, and the consequent cost of evaporation and make-up chemical may be too high for practical use on a commercial scale. It is the purpose of this presentation to evaluate the quantities involved, the evaporation necessary for recovery of chemical, and the chemical loss in washing the pulp.

Briefly, the pulping process is as follows. Before cooking, the chips are subjected to the mechanical action of a hammer mill or attrition mill, splitting them along the grain, avoiding cross-grain breakage. This splitting of the chips is essential in order that maximum wood surface may be exposed to the action of the cooking chemical, permitting uniform pulping at high speed. Chips may be presteamed for softening purposes before being subjected to the mechanical subdivision, or they may be split as they come from the chip screens, without presteaming.

The shredded chips are fed to a continuous digester where they are mixed with the cooking liquor, a mixture of highly concentrated white liquor and dilute black liquor from the washing system. The concentration of the liquor in contact with the chips is critical. High concentrations of liquor, in the range of 80 grams $Na_2O$ per liter, will produce pulps with only 2.0 to 3.0% pentosan, while low concentrations, 20 grams per liter or less, result in higher pentosan and consequently higher strength pulps. The concentration of the cooking liquor will depend on the end use of the pulp, whether for dissolving grades or high strength pulps.

In order to keep the reaction rate high, concentration of chemical is maintained by fortification with high concentration white liquor at several points along the digester. When concentration drops to 10% below the desired average, fortification liquor is added to bring the concentration to a value 10% higher than the average. Contact of the wood with this highly concentrated fortification liquor (about 32% $Na_2O$) is avoided by mixing the fortification liquor with constantly recirculated black liquor from the digester discharge.

It is possible to carry out the cook at constant steam pressure since a continuous digester is used. The steam pressure used will depend on the average concentration of the cooking liquor. High speed reaction is maintained by using higher steam pressures when the lower concentrations are employed. Generally, when concentration of cooking liquor is 20 grams per liter, steam pressure is maintained at 170 to 190 p.s.i.g. for a full chemical cook. At concentrations of 80 grams per liter, steam pressures are held at 135 to 160 p.s.i.g. By proper adjustment of these two variables, fully cooked pulps are produced in 10 to 20 min.

The problem of determining quantity of chemical needed in cooking and evaporation load necessary for the recovery system requires that the digester system and the washing system be considered simultaneously. The two systems are mutually dependent. The amount of white liquor required by the digester depends on the quantity and concentration of the dilute black liquor returned from the washing system. The concentration and amount of black liquor, which the washing system sends to the evaporators, depend on the total solids in the liquor which the digester system sends to the washing system. Therefore, a method of calculation must be set up which involves both systems.

### ANALYSIS OF THE PROBLEM

The problem can be visualized with the aid of Figs. 1 and 2. Figure 1 is a schematic presentation of a continuous digester while Fig. 2 represents a four-stage screw press washing system. The screw press method of washing is used here because it lends itself readily to mathematical analysis of quantities involved better than a vacuum washer system. Vacuum washers will be considered later in the calculations.

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In the digester system (Fig. 1), the quantity of wood and its moisture (50%) are fixed. Concentration of white liquor is fixed at 32% $Na_2O$ but the quantity is unknown, depending on the amount of $Na_2O$ which enters with the dilute black liquor. The quantity and concentration of dilute black liquor are not known, the quantity depending on the liquor ratio and the amount of white liquor. The concentration of alkali

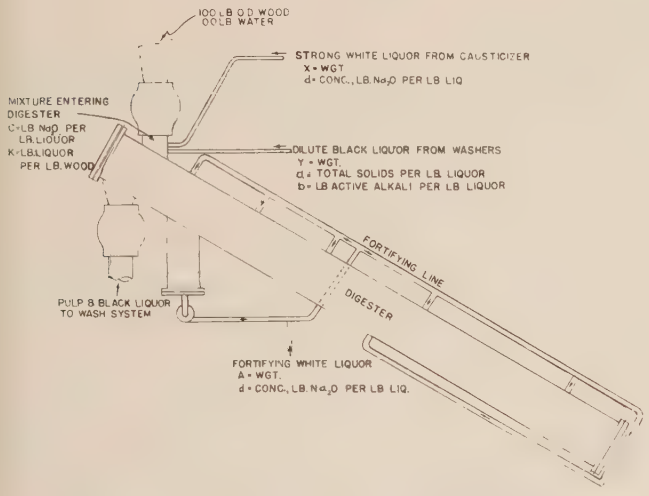
100 LB O.D. WOOD
100 LB WATER
STRONG WHITE LIQUOR FROM CAUSTICIZER
X = WGT.
d = CONC., LB. Na2O PER LB LIQ
MIXTURE ENTERING DIGESTER
C = LB NaO PER LB. LIQUOR
K = LB. LIQUOR PER LB. WOOD
DILUTE BLACK LIQUOR FROM WASHERS
Y = WGT.
d = TOTAL SOLIDS PER LB. LIQUOR
b = LB. ACTIVE ALKALI PER LB. LIQUOR
FORTIFYING LINE
DIGESTER
PULP & BLACK LIQUOR TO WASH SYSTEM
FORTIFYING WHITE LIQUOR
A = WGT.
d = CONC., LB. Na2O PER LB LIQ.

*Fig. 1. Flow sheet for continuous pulping*

in this dilute black liquor depends on the strength of the black liquor leaving the digester and the operating variables in the washing system.

It is possible to set up a series of equations for the digester system when the cooking conditions are fixed. For example, if the liquor ratio is fixed at 3.5 lb. of mixed liquor per lb. of dry chip and the weight of wood is fixed at 100 lb. moisture-free chips, an equation can be written involving the weights of white liquor and dilute black liquor.

As soon as the starting concentration is fixed at, say, 88 grams $Na_2O$ per liter, it is possible to set up another equation involving both quantity and concentration of white liquor and of dilute black liquor.

When yield of pulp is fixed, and the chemical used to attain this yield is known, another relationship between white liquor, dilute black liquor, and fortification liquor is set up. Care must be exercised in fixing the yield to be sure that the yield chosen is substantiated by actual cooking data for the conditions specified. A chemical consumption figure of 0.25 lb. of $Na_2O$ consumed per lb. of woody material dissolved is used in these calculations. This figure has been verified by experimental data (*3*).

The total weight of black liquor can be expressed in terms of the liquor ratio, fortification liquor, and wood dissolved. Its concentration of active alkali and total solids can also be expressed in terms of the various streams.

When all of the equations have been written it is possible to develop a relationship between total solids in the dilute black liquor entering the digester and in the strong black liquor leaving the digester. This relationship, being derived from the equations, satisfies the conditions imposed by digester operation but is independent of the washing system. The relationship can be expressed graphically when calculated values for total solids in the dilute black liquor are plotted against assumed values for total solids in the strong black liquor.

A similar relationship can be developed for the washing system. Referring to Fig. 2, if $X_1$ is taken as yield of pulp and $Y_1$ is assigned as the weight of total black liquor imposed by a particular set of digester operating conditions, a relationship can be developed between $C_1$ and $C_2$. It is apparent from the figure that $C_1$ corresponds to total solids in the black liquor leaving the digester and $C_2$ corresponds to the total solids in the dilute black liquor returning to the digester. The relationship between $C_1$ and $C_2$ is primarily controlled by the consistency of feed to each of the presses, the consistency of pulp discharge from each press, and the number of presses in series. The calculated values of $C_2$ can be plotted graphically against assumed values for $C_1$ on the same chart containing the digester relationship. The point of intersection of the two lines defines the only value of total solids in each black liquor stream which satisfies both the digester and washing conditions imposed.

Once this point of intersection has been fixed it is possible to calculate, by use of the appropriate digester equations, the quantities of white liquor, dilute black liquor, and fortification liquor needed. The amount of strong black liquor going to the evaporator and its per cent total solids becomes known. The quantity of dilute liquor to the evaporator can be computed, subtracting from the total amount that quantity which is returned to the digester. The total evaporation can then be calculated for each set of conditions, assuming that in all cases evaporation is carried out to 60% solids.

It is necessary to explore the two extremes of continuous pulping. In one case, concentration of alkali is held at a high figure to insure that the pentosan content of the pulp will be at a low value (2.0 to 3.0%) suitable for dissolving purposes. On the other hand, concentration of alkali is kept as low as possible to insure high pentosan content of the pulps for papermaking grades.

In the high concentration range three modifications

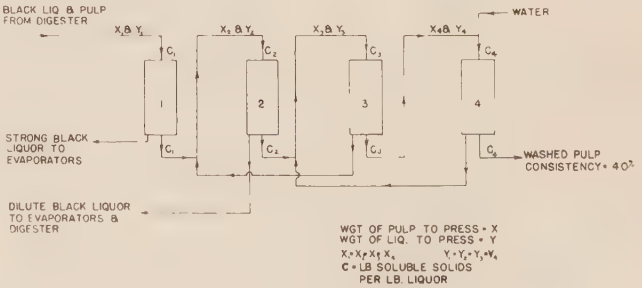
BLACK LIQ. & PULP FROM DIGESTER
WATER
STRONG BLACK LIQUOR TO EVAPORATORS
WASHED PULP CONSISTENCY = 40%
DILUTE BLACK LIQUOR TO EVAPORATORS & DIGESTER
WGT OF PULP TO PRESS = X
WGT OF LIQ. TO PRESS = Y
C = LB SOLUBLE SOLIDS PER LB. LIQUOR

*Fig. 2. Four-stage screw press washing system*

were studied. In the first, average concentration throughout the cook was held at a figure of 80 grams $Na_2O$ per liter. Maximum was 88 and minimum was 72 grams per liter. The final yield was 43%. In the second variation, the average concentration of 80 grams per liter was held only until 40% of the wood was dissolved, at which point the concentration was 80 grams per liter. No further fortification of liquor

was used, the concentration being allowed to fall until the final yield of 43% was realized. The third modification was exactly like the second with the exception that fortification was cut off slightly sooner so that the concentration at 40% wood removal was 72 grams per liter. This falling concentration in the latter part of cook for production of low pentosan pulps has not been carried out experimentally. However, experimental data (*2*) indicate that pentosan is removed rather early in the cook, so it seems safe to assume that pentosan removal will be completed by the time the cook has progressed to 40/57 or 70% of completion.

In low concentration pulping, two variations in liquor concentration were studied. In one case, the concentration was held at an average of 20 grams per liter (max. 22; min. 18) throughout the cook until the final yield of 48% was reached. In the second case, to reduce the amount of fortification liquor, the initial concentration was set at 50 grams per liter and allowed to fall to a value of 20 grams per liter before fortification was used to hold the concentration for the rest of the cook at an average of 20 grams per liter. This variation seems reasonable because concentration will fall at a very rapid rate to 20 grams per liter but should be verified by experimental data to insure that pentosan content of the pulp is satisfactory.

Several different liquor-wood ratios were investigated for each condition of concentration. In all cases ratios of 2.5 and 3.5 were used and, in two instances, a liquor-wood ratio as low as 2.0 was used.

Three separate washing systems were studied in combination with the various cooking conditions. The first two systems involved the use of four screw presses in series, the third system used three vacuum washers in series.

In the first screw press washing system, the first press was fed with pulp at the consistency leaving the digester. The pulp, discharged from each press at 40% consistency, was diluted to the same consistency as the original feed before being fed to the next press in series.

In the second system, initial feed to the first press and consistency of discharge from each press was the same as in the first system. The pulp discharge of 40% was diluted to 15% before being fed to the next press in series.

The third system used three stages of vacuum washers, similar to Fig. 3. The only numerical values used from the figure were the concentrations of 16.1 and 22.0% total solids in the black liquor feed and filtrate, respectively.

## CALCULATIONS

It was first necessary to calculate the amount of chemical which must be added as fortification liquor to meet the conditions set for each type cook.

Conditions are as follows:

1. Initial concentration (including water in chips), 88 grams $Na_2O$ per liter; average concentration throughout, 80 grams per liter (max. 88; min. 72 g.); final concentration at a yield of 43%, 72 grams per liter.
2. Initial concentration, 88 grams per liter; average concentration held at 80 grams per liter until yield is 60%, at which point concentration is 80 grams per liter; concentration allowed to fall thereafter without any further addition of chemical until final yield is 43%.
3. All conditions the same as (2) except that concentration is 72 grams $Na_2O$ per liter when yield is 60%.
4. Initial concentration, 50 grams $Na_2O$ per liter; concentration allowed to drop to 20 grams per liter; held at an average value of 20 grams per liter (max. 22; min. 18) until final yield is 48%; final concentration is 18 grams per liter.

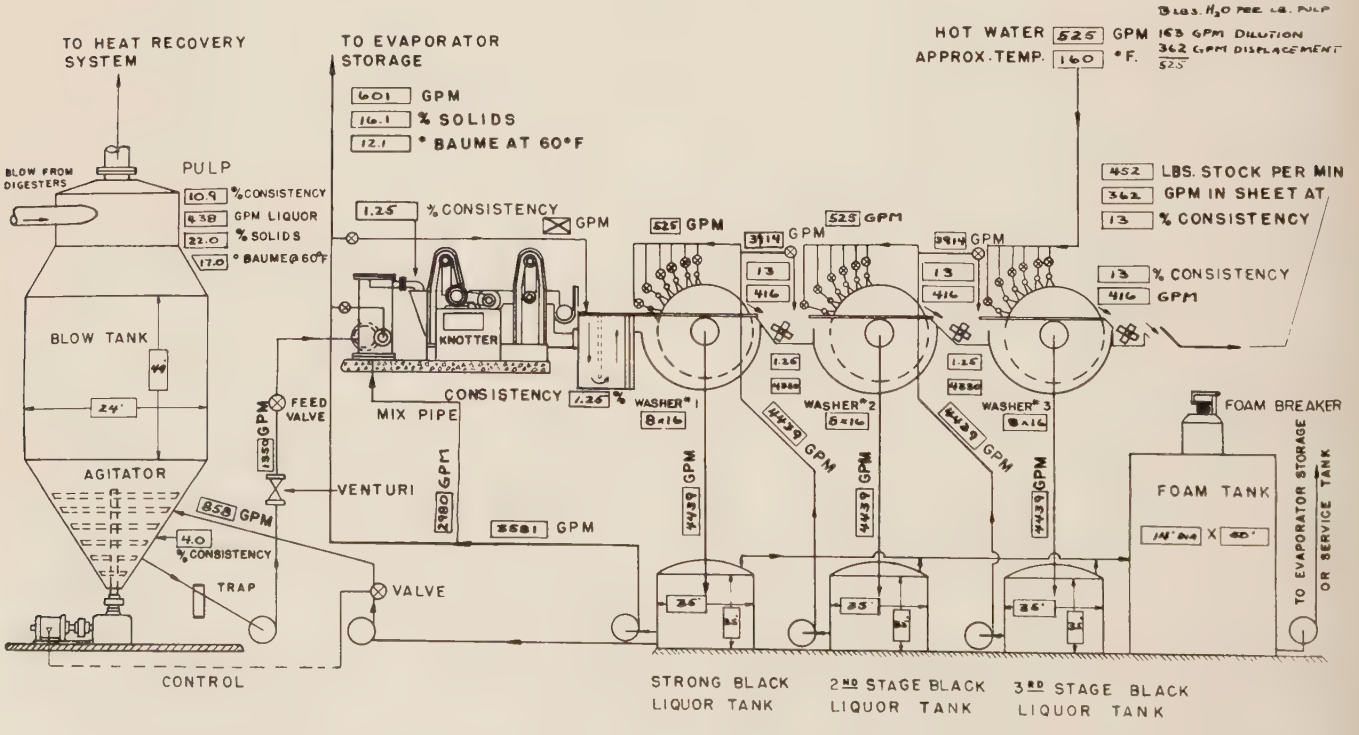


*Fig. 3. Three-stage brown stock washing system flow diagram*

| Cooking conditions | Initial g./l. | Concentration Sp. gr. | Concentration % | Yield, % of original wood | Concentration at specified yield G./l. | Concentration at specified yield Sp. gr. | Concentration at specified yield % |
|---|---|---|---|---|---|---|---|
| 1 | 88 | 1.110 | 7.91 | 43 | 72 | 1.090 | 6.59 |
| 2 | 88 | 1.110 | 7.91 | 60[a] | 80 | 1.105 | 7.24 |
| 3 | 88 | 1.110 | 7.91 | 60[a] | 72 | 1.090 | 6.59 |
| 4 | 50 | 1.080 | 4.62 | 48 | 18 | 1.060 | 1.695 |
| 5 | 22 | 1.060 | 2.07 | 48 | 18 | 1.060 | 1.695 |

[a] These figures are not final yield values, but signify the point in the reaction beyond which concentration is allowed to fall with no further fortification of chemical.

5. Initial concentration, 22 grams per liter; held at an average of 20 grams per liter until the final yield is 48%; final concentration, 18 grams per liter.

The amount of fortifying chemical can be calculated from the initial and final concentrations, liquor-wood ratio, pulp yield and chemical consumption per unit weight of woody material dissolved. The concentrations for the conditions specified must be, for calculation purposes, expressed as per cent of the liquor rather than as grams per liter and are tabulated above.

The method for calculating the quantity of fortifying liquor for a specified set of conditions is as follows:

Basis: 100 lb. moisture-free wood
Initial concentration = 88 grams per liter or 7.91% $Na_2O$
Wood moisture = 50%
Liquor wood ratio = 3.5
Concentration of fortifying liquor = 32.0% $Na_2O$
Final concentration = 72 grams per liter or 6.59% $Na_2O$
Final yield of pulp = 43% of original wood
Total weight of liquor at start of cook = $100 \times 3.5 = 350$ lb.
Weight of $Na_2O$ added at start = $350 \times 0.0791 = 27.7$ lb.
Additional $Na_2O$ added by fortification = $0.32 \times$ weight fortification liquor
Weight of $Na_2O$ consumed = $0.25\,(100 - 43) = 14.25$ lb.
Weight of black liquor = $350 + 57 +$ weight of fortification liquor
Concentration of alkali in black liquor = 6.59% or 0.0659 =

$$\frac{350 \times 0.0791 + 0.32 \times \text{fortification liquor} - 0.25 \times 57}{350 + 57 + \text{fortification liquor}}$$

weight of fortification liquor = 52.70 lb.

The results of the calculations for all conditions are listed in Table I.

**Table I. Fortification Liquor Required by Various Cooking Conditions**

(Based on 100 lb. moisture-free wood containing 50% moisture)

| Cooking conditions | Liquor-wood ratio | Weight of fortifying liquor, lb. | Fortifying chemical, lb. $Na_2O$ |
|---|---|---|---|
| (1) 88 g./l. at start, av. of 80 g./l. throughout, final yield is 43% | 2.5 | 57.9 | 18.52 |
| | 3.5 | 52.7 | 16.87 |
| (2) 88 g./l. at start, av. conc. 80 g./l. until yield is 60%. At this point conc. is 80 g./l. | 2.0 | 46.8 | 14.96 |
| | 2.5 | 45.3 | 14.50 |
| | 3.5 | 42.6 | 13.63 |
| (3) 88 g./l. at start, av. conc. 80 g./l. until yield is 60%. At this point conc. is 72 | 2.5 | 36.6 | 11.74 |
| | 3.5 | 31.6 | 8.42 |
| (4) 50 g./l. at start, drop to 20, held at average of 20 g./l. until yield is 48%. Conc. at 48% yield is 18 g./l. | 2 | 26.5 | 8.48 |
| | 2.5 | 21.7 | 6.95 |
| | 3.5 | 12.1 | 3.87 |
| (5) 22 g./l. at start, held at av. of 20 g./l. throughout the cook Final yield = 48% Conc. at end = 18 g./l. | 2.5 | 42.8 | 13.70 |
| | 3.5 | 41.6 | 13.30 |

The concentrations and amounts of white and black liquor required for each set of cooking conditions must be evaluated. To do this it is necessary to set up a series of simultaneous equations containing the variables involved. The nomenclature for the various quantities is as follows.

**Nomenclature**

A.A. = active alkali
B.L. = black liquor
T.S. = total solids
$x$ = weight of white liquor mixed with the chips
$A$ = weight of white liquor added as fortifying liquor
$y$ = weight of dilute black liquor recirculated to the digester from the recovery system
$d$ = concentration of active alkali in white liquor (pounds $Na_2O$ per pound liquid)
$a$ = concentration of total solids in dilute black liquor (pounds T.S. per pound liquid)
$b$ = concentration of active alkali in dilute black liquor (pounds $Na_2O$ per pound liquid)
$c$ = concentration of active alkali in mixed cooking liquor (pounds $Na_2O$ per pound liquid, including water in chips)
$K$ = liquor wood ratio (pounds mixed liquor per pound moisture-free wood)
$P$ = weight of wood dissolved during the cook per 100 lb. wood charged

## DERIVATION OF DIGESTER EQUATIONS

Basis of calculations: 100 lb. moisture-free wood charged
The total liquid charged to the digester with the chips may be expressed as

$$x + y + 100 = 100K \qquad (1)$$

The total active alkali charged to the digester entrance is

$$dx + by = 100Kc \qquad (2)$$

The concentration of total solids in the black liquor leaving the digester, expressed as pounds of total solids per pound of black liquor is

$$\frac{dx + dA + ay + P}{100K + A + P} = \text{lb. T.S./lb. B.L.} \qquad (3)$$

The ratio of active alkali to total solids in the exit black liquor is

$$\frac{dx + dA + by - 0.25P}{dx + dA + ay + P} = \text{lb. A.A./lb. T.S.} \qquad (4)$$

Eliminating $x$ from equation (3) by substituting its value in equation (1), equation 3 becomes

$$\frac{100d\,(K - 1) - dy + dA + ay + P}{100K + A + P} = \frac{100d(K - 1) + dA + P - y\,(d - a)}{100K + A + P} = \text{lb. T.S./lb. B.L.} \qquad (5)$$

By a similar elimination of $x$, equation (4) becomes

$$\frac{d(100\,[K - 1] - y) + dA + by - 0.25P}{d(100\,[K - 1] - y) + dA + ay + P} = \frac{100d\,(K - 1) + dA - 0.25P - y(d - b)}{100d\,(K - 1) + dA + P - y(d - a)} = \frac{\text{lb. A.A.}}{\text{lb. T.S.}} \qquad (6)$$

An expression for $y(d - b)$ may be found from equation (2) as follows:

$$d(100K - 100 - y) + by = 100Kc$$
$$100d(K - 1) - dy + by = 100Kc$$
$$y(d - b) = 100d(K - 1) - 100Kc \quad (7)$$

this value for $y(d - b)$ may be substituted in equation (6), as follows:

$$\frac{100d(K - 1) + dA - 0.25P - 100d(K - 1) + 100Kc}{100d(K - 1) + dA + P - y(d - a)} =$$
$$\frac{100Kc + dA - 25P}{100d(k - 1) + dA + P - y(d - a)} = \text{lb. A.A./lb. T.S.} \quad (8)$$

The ratio of active alkali to total solids in the black liquor, as expressed by equation (8), must be numerically equal to the similar ratio of active alkali to total solids in the dilute black liquor recirculated from the washing system. This being the case, equation (8) becomes

$$\frac{100Kc + dA - 0.25P}{100d(k - 1) + dA + P - y(d - a)} = \frac{b}{a} \quad (9)$$

Equations (5), (7), and (9) can be used to evaluate the various quantities when the digester conditions are set. The absolute values for these quantities cannot be set until the degree of dilution resulting from the washing system is known. This dilution will, of course, have its effect on the other digester streams. However, it is possible to assume a definite value for the total solids in the black liquor and, from the three equations, calculate the absolute values of all quantities which correspond to the assumed values of black liquor solids.

For example, let it be stated that the digester will be operated under such conditions that the average concentration throughout the cook will be 80 grams $Na_2O$ per liter. Concentration will be maintained by means of fortification with white liquor containing 32% $Na_2O$. The concentration may vary between 88 and 72 grams per liter or ±10%. Therefore, initial concentration is 88 grams per liter and final concentration is 72 grams per liter. The liquor ratio will be 3.5 lb. mixed liquor per lb. moisture-free wood. Final yield will be 43% of the wood charged. Under these conditions the digester constants are as follows:

$K = 3.5$
$A$ (from Table I) $= 52.7$
$P = 100 - 43 = 57$
$c = 0.0791$ (88 g./l.)
$d = 0.32$
$a$, $b$, $y$, and $x$ are unknown
From equation (7),

$$y(0.32 - b) = 32 \times 2.5 - 350 \times 0.0791$$
$$y = \frac{80 - 27.7}{0.32 - b} = \frac{52.3}{0.32 - b} \quad (10)$$

From equation (5),

$$\frac{\text{Pounds T.S.}}{\text{Pounds B.L.}} = \frac{32 \times 2.5 + 0.32 \times 52.7 + 57 - y(0.32 - a)}{350 + 52.7 + 57}$$
$$= \frac{80 + 16.85 + 57 - y(0.32 - a)}{459.7}$$
$$= \frac{153.85 - y(0.32 - a)}{459.7} \quad (11)$$

From equation (9),

$$\frac{b}{a} = \frac{350 \times 0.0791 + 0.32 \times 52.7 - 0.25 \times 57}{153.85 - y(0.32 - a)}$$
$$= \frac{27.7 + 16.85 - 0.425}{153.85 - y(0.32 - a)}$$
$$= \frac{30.30}{153.85 - y(0.32 - a)}$$

or, using equation (11),

$$= \frac{30.30}{459.7 \times \text{T.S. in B.L.}} \quad (12)$$

Assume the total solids in the black liquor to be 30%,

$$\frac{b}{a} = \frac{30.30}{459.7 \times 0.30} = \frac{30.30}{138} = 0.219$$

From equation (11),

$$0.30 \times 459.7 = 153.85 - y(0.32 - a)$$
$$y = \frac{153.85 - 138}{0.32 - a} = \frac{15.85}{0.32 - a}$$

therefore, from equation (10)

$$\frac{52.3}{0.32 - b} = \frac{15.85}{0.32 - a}$$
$$52.3 \times 0.32 - 52.3a = 15.85 \times 0.32 - 0.219 \times 15.85a$$
$$(52.3 - 3.47)a = (52.3 - 15.85) \times 0.32$$
$$a = \frac{36.45 \times 0.32}{48.83} = 0.239$$
$$b = 0.239 \times 0.219 = 0.0524$$
$$y = \frac{15.85}{0.32 - 0.239} = 195.5$$
$$y = \frac{52.3}{0.32 - 0.524} = 195.5$$
$$x = 100 \times 3.5 - 100 - y = 250 - 195.5 = 54.5$$

Assumption of various values for total solids in the black liquor results in the following relationships.

**Table II. Relationship Between Total Solids in Black Liquor Discharge and in Recirculated Black Liquor**

| *T.S. in B.L., %* | a | b | y | x |
|---|---|---|---|---|
| 35 | 0.3545 | 0.0667 | 207 | 43 |
| 30 | 0.2390 | 0.0524 | 196 | 54 |
| 26 | 0.1325 | 0.0335 | 183 | 67 |
| 24 | 0.0680 | 0.0190 | 174 | 76 |
| 22 | −0.0062 | −0.0089 | 163 | 87 |

Values of $a$, $b$, $y$, and $x$ for several assumed values of total solids in the black liquor for each of the cooking conditions specified in Table I were calculated as above. These calculations are listed in Table III. These relationships show that, under the cooking conditions specified, for any concentration of black liquor leaving the digester, the quantity and analysis of the recirculating black liquor and the quantity of white liquor are fixed. It now remains to find another relationship between the quantities and analyses of these two liquors. They can then be handled simultaneously to find values which satisfy both the digestion and the washing systems. Such a relationship can be found in the brown stock washing system.

### *Brown Stock Washing*

*Screw Press Washing.* Since screw press washers lend themselves more readily to mathematical analysis than vacuum washers, a four-stage screw press installation will be analyzed to develop this relationship. Consider the four-stage washing system shown in Fig. 2.

Let $X$ = pounds of moisture-free pulp per 100 lb. dry wood to digester

$Y$ = lb. black liquor fed with pulp to each stage

$c$ = fraction of total solids in the black liquor

Subscripts 1, 2, 3, and 4 refer to the stage of pressing.

$$X_1 = X_2 = X_3 = X_4$$

$$Y_1 = Y_2 = Y_3 = Y_4$$

from a soluble solids balance on stage four

$$\frac{60}{40} X c_3 = \frac{60}{40} X c_4 + \left(Y - \frac{60}{40} X\right) c_4$$

$$1.5Xc_3 = 1.5Xc_4 + Yc_4 - 1.5Xc_4$$

$$1.5Xc_3 = Yc_4$$

$$c_3 = \frac{Y}{1.5X} c_4 \qquad (13)$$

From a soluble solids balance on stage three

$$1.5Xc_2 + (Y - 1.5X)c_4 = Yc_3$$

$$= Y \frac{Y}{1.5X} c_4 = \frac{Y^2}{1.5X} c_4$$

$$1.5Xc_2 = \frac{Y^2}{1.5X} c_4 - Yc_4 + 1.5Xc_4$$

$$C_2 = \frac{1}{1.5X} c_4 \left(\frac{Y^2}{1.5X} - Y + 1.5X\right)$$

$$= c_4 \left[\left(\frac{Y}{1.5X}\right)^2 - \frac{Y}{1.5X} + 1\right]$$

$$= c_4 \left[\left(\frac{Y}{1.5X} - 0.50\right)^2 + 0.75\right] \qquad (14)$$

From a soluble solids balance on stage two

$$1.5Xc_1 + (Y - 1.5X)c_3 = Yc_2$$

Substitute equation (13) for $c_3$ and equation (14) for $c_2$

$$1.5Xc_1 + (Y - 1.5X) \frac{Y}{1.5X} c_4 = Yc_4 \left[\left(\frac{Y}{1.5X} - 0.50\right)^2 + 0.75\right]$$

expanding

$$1.5Xc_1 + \left(\frac{Y^2}{1.5X} - Y\right) c_4 = Yc_4 \left[\left(\frac{Y}{1.5X}\right)^2 - \frac{Y}{1.5X} + 1\right]$$

**Table III. Quantities and Concentrations of Black Liquors**

(Continuous cooking)

| *Cooking conditions* | K | C | A | P | *T.S. per lb. B.L.* | a | b | y |
|---|---|---|---|---|---|---|---|---|
| 88 grams per liter at start. Av. of 80 grams per liter throughout. Final yield is 43% with final concentration at 72 grams per liter. | 2.5 | 0.0791 | 57.9 | 57 | 0.35 | 0.3582 | 0.0691 | 112 |
| | | | | | 0.325 | 0.2738 | 0.0580 | 107 |
| | | | | | 0.30 | 0.1803 | 0.0405 | 101 |
| | | | | | 0.285 | 0.1126 | 0.0267 | 95 |
| | | | | | 0.27 | 0.0467 | 0.0117 | 92 |
| | 3.5 | 0.0791 | 52.7 | 57 | 0.35 | 0.3546 | 0.0667 | 207 |
| | | | | | 0 30 | 0 2390 | 0.0524 | 196 |
| | | | | | 0.26 | 0.1325 | 0.0335 | 183 |
| | | | | | 0.24 | 0.0680 | 0.0190 | 174 |
| | | | | | 0.22 | −0.0062 | −0 0089 | 163 |
| 88 grams per liter at start. Av. of 80 grams per liter until yield is 60%. At this point concentration is 80 grams per liter. No further fortification thereafter. Final yield is 43%. | 2.0 | 0.0791 | 46.8 | 57 | 0.35 | 0.3580 | 0.0560 | 62 |
| | | | | | 0.31 | 0.1415 | 0.0249 | 55 |
| | | | | | 0.30 | 0.0771 | 0.0140 | 53 |
| | | | | | 0.29 | 0.0085 | 0.0016 | 51 |
| | 2.5 | 0.0791 | 45.3 | 57 | 0.35 | 0.3553 | 0 0577 | 108 |
| | | | | | 0.30 | 0.1797 | 0.0339 | 98 |
| | | | | | 0.27 | 0 0540 | 0.0114 | 92 |
| | | | | | 0.25 | −0.0486 | −0.0110 | 85 |
| | 3.5 | 0.0791 | 42.6 | 57 | 0.35 | 0.352 | 0.0607 | 202 |
| | | | | | 0.30 | 0.239 | 0 0478 | 192 |
| | | | | | 0.26 | 0.134 | 0.0310 | 181 |
| | | | | | 0.24 | 0.074 | 0.0186 | 174 |
| | | | | | 0.22 | 0.005 | 0 0014 | 164 |
| 88 grams per liter at start. Av. of 80 grams per liter until yield is 60%. At this point concentration is 72 grams per liter. No further fortification thereafter. | 2.5 | 0.0791 | 36.7 | 57 | 0.35 | 0.354 | 0.059 | 106 |
| | | | | | 0.30 | 0.180 | 0.030 | 97 |
| | | | | | 0.27 | 0.0587 | 0.011 | 91 |
| | | | | | 0.25 | −0.0381 | −0.0077 | 86 |
| | 3 5 | 0.0791 | 26.3 | 57 | 0.35 | 0.352 | 0.054 | 197 |
| | | | | | 0.30 | 0.237 | 0.042 | 188 |
| | | | | | 0.26 | 0.135 | 0.028 | 179 |
| | | | | | 0.24 | 0.0774 | 0.0173 | 173 |
| | | | | | 0.22 | 0.0136 | 0.0033 | 165 |
| 50 grams per liter at start. Concentration allowed to drop to 20 grams per liter without fortification. Concentration held at av. of 20 grams per liter by fortification until final yield is 48%. Concentration at this point is 18 grams per liter. | 2.0 | 0.0462 | 26.5 | 52 | 0.32 | 0.274 | 0.0142 | 74 |
| | | | | | 0.30 | 0.199 | 0.0111 | 74 |
| | | | | | 0.28 | 0.121 | 0 0072 | 73 |
| | | | | | 0.26 | 0 0399 | 0.0026 | 72 |
| | 2.5 | 0.0462 | 21.7 | 52 | 0.30 | 0.238 | 0.0132 | 119 |
| | | | | | 0.275 | 0.1695 | 0.0102 | 118 |
| | | | | | 0.25 | 0.095 | 0.0064 | 116 |
| | | | | | 0.22 | 0.0075 | 0.0006 | 114 |
| | 3.5 | 0.0462 | 12.1 | 52 | 0.35 | 0.361 | 0.0172 | 210 |
| | | | | | 0.30 | 0.263 | 0.0146 | 209 |
| | | | | | 0.275 | 0.215 | 0.013 | 208 |
| | | | | | 0.25 | 0.164 | 0.0109 | 206 |
| | | | | | 0 20 | 0.0589 | 0.00489 | 202 |
| 22 grams per liter at start. Av. of 20 grams per liter until final yield of 48% is reached. Concentration at this point is 18 grams per liter | 2.5 | 0.0207 | 42.8 | 52 | 0.35 | 0.364 | 0.0178 | 142 |
| | | | | | 0.30 | 0.245 | 0.0140 | 140 |
| | | | | | 0.275 | 0.182 | 0.0113 | 139 |
| | | | | | 0.25 | 0.118 | 0.008 | 137 |
| | 3.5 | 0.0207 | 41.6 | 52 | 0 35 | 0.359 | 0.0176 | 241 |
| | | | | | 0.30 | 0.267 | 0.0153 | 239 |
| | | | | | 0.275 | 0.221 | 0.0138 | 238 |
| | | | | | 0.25 | 0.175 | 0.0119 | 236 |
| | | | | | 0.20 | 0.075 | 0.0064 | 232 |

$d$ = 0.32 lb. $Na_2O$ per lb. white liquor.

$$1.5Xc_1 = c_4\left[\frac{Y^3}{(1.5X)^2} - \frac{Y^2}{1.5X} + Y - \frac{Y^2}{1.5X} + Y\right]$$

$$= c_4\left[\frac{Y^3}{1.5X^2} - \frac{2Y^2}{1.5X} + 2Y\right]$$

$$c_1 = c_4\left[\left(\frac{Y}{1.5X}\right)^3 - 2\left(\frac{Y}{1.5X}\right)^2 + 2\frac{Y}{1.5X}\right]$$

$$= c_4\left[\left(\frac{Y}{1.5X} - 1\right)^3 + \left(\frac{Y}{1.5X} - 0.50\right)^2 + 0.75\right] \quad (15)$$

By use of equations (14) and (15), values of $c_2$ can be calculated which correspond to assumed values of $c_1$. $X$ and $Y$ are, in reality, the weight of pulp and weight of black liquor, respectively, discharged from the digester for every 100 lb. dry wood charged to the digester. The numerical values for $X$ and $Y$ can be calculated for each set of digester conditions described previously. Under these conditions, $c_1$ becomes the fraction of total solids in the black liquor. $c_2$ is then the fraction of total solids in the dilute black liquor which the washing system will produce. The following sample calculation shows how the values of $c_2$ are calculated for assumed value of $c_1$.

Using the digester conditions used previously for the calculation of digester quantities:

$K = 3.5$
$A = 52.7$
$P = 57$
Yield = 43 lb.
Therefore $X = 43$

$$Y = 350 + 52.7 + 57 = 459.7$$

$$\frac{Y}{1.5X} = \frac{460}{1.5 \times 43} = 7.13$$

$$\left(\frac{Y}{1.5X} - 1\right)^3 = (6.13)^3 = 230$$

$$\left(\frac{Y}{1.5X} - 0.5\right)^2 = (6.63)^2 = 44.0$$

when $c_1 = 0.35$, from equation (15)

$$0.35 = (230 + 44.0 + 0.75)c_4$$
$$= 274.75c_4$$

$$c_4 = \frac{0.35}{274.75} = 0.001275$$

from equation (14)

$$c_2 = c_4(44.0 + 0.75)$$
$$= 0.001275 \times 44.75 = 0.0570$$

when $c_1 = 0.30$

$$c_2 = \frac{0.30 \times 44.75}{274.75} = 0.0488$$

when $c_1 = 0.26$

$$c_2 = 0.26 \times \frac{44.75}{274.75} = 0.0423$$

when $c_1 = 0.24$

$$c_2 = 0.24 \times \frac{44.75}{274.75} = 0.0391$$

when $c_1 = 0.22$

$$c_2 = 0.22 \times \frac{44.75}{274.75} = 0.0356$$

Table IV lists the values of necessary constants and the calculated values of $c_2$ for assumed value of $c_1$ under all cooking conditions.

Thus, two independent relationships have been developed between the total solids in the black liquor discharged from the digester and the total solids in the dilute black liquor discharged from the washing system and returned for recirculation in the digester. The first of these relationships, listed in Table II, is controlled by the conditions of concentration and yield specified by the digester. The second relationship is entirely controlled by the washing conditions. These two relationships are listed in Table V.

It can be seen from Table V that if the per cent total solids in the strong black liquor, as discharged from the

**Table IV. Calculation of Weak Liquor Concentration in Four-State Press Washing**

(Consistency of feed to all presses constant at consistency of digester discharge)

| *Cooking conditions* | K | Y | $\left(\frac{Y}{1.5X} - 1\right)^3$ | $\left(\frac{Y}{1.5X} - 0.5\right)^2$ | $C_1$ | *Factor*[a] | $C_2$ |
|---|---|---|---|---|---|---|---|
| See Fig. 4 | 2.5 | 364.9 | 101 | 26.5 | 0.20 | 0.212 | 0.0425 |
| | | | | | 0.35 | 0.212 | 0.0743 |
| | 3.5 | 469.7 | 249 | 45.8 | 0.20 | 0.157 | 0.0314 |
| | | | | | 0.25 | 0.157 | 0.0550 |
| See Fig. 5 | 2.0 | 303.8 | 51.2 | 17.7 | 0.20 | 0.265 | 0.0530 |
| | | | | | 0.35 | 0.265 | 0.0928 |
| | 2.5 | 352.3 | 89 | 25.2 | 0.20 | 0.221 | 0.0443 |
| | | | | | 0.35 | 0.221 | 0.0775 |
| | 3.5 | 449.6 | 214 | 41.8 | 0.20 | 0.165 | 0.0350 |
| | | | | | 0.35 | 0.165 | 0.0578 |
| See Fig. 6 | 2.5 | 343.6 | 81 | 23.1 | 0.20 | 0.227 | 0.0455 |
| | | | | | 0.35 | 0.227 | 0.0795 |
| | 3.5 | 438.6 | 195 | 39.5 | 0.20 | 0.171 | 0.0342 |
| | | | | | 0.35 | 0 171 | 0.0599 |
| See Fig. 7 | 2.0 | 278.5 | 23.4 | 11.3 | 0.20 | 0.340 | 0.0680 |
| | | | | | 0.35 | 0.340 | 0.1190 |
| | 2.5 | 323.7 | 42.9 | 16.0 | 0.20 | 0.281 | 0.0563 |
| | | | | | 0.35 | 0.281 | 0.0984 |
| | 3.5 | 414.1 | 106.5 | 27.5 | 0.20 | 0.210 | 0.0420 |
| | | | | | 0.35 | 0.210 | 0.0734 |
| See Fig. 8 | 2.5 | 344.8 | 55 | 18.4 | 0.20 | 0.258 | 0.0517 |
| | | | | | 0.35 | 0.258 | 0.0907 |
| | 3.5 | 443.6 | 137 | 32 | 0.20 | 0.258 | 0.0386 |
| | | | | | 0.35 | 0.193 | 0.0675 |

[a] Factor = $\dfrac{\left(\frac{Y}{1.5} - 0.5\right)^2 + 0.75}{\left(\frac{Y}{1.5x} - 1.0\right)^3 + \left(\frac{Y}{1.5x} - 0.5\right)^2 + 0.75}$.

**Table V. Relationship Between Total Solids Content of Black Liquor**

| *Black liquor from digester or to washing system (lb. T.S./lb. B.L.)* | *Dilute black liquor (lb. T.S./lb. B.L.) From washer calculations* | *From digester calculations* |
|---|---|---|
| 35 | 0.0570 | 0.3545 |
| 30 | 0.0488 | 0.2390 |
| 26 | 0.0423 | 0.1325 |
| 24 | 0.0391 | 0.0680 |
| 22 | 0.0356 | −0.0062 |

digester and fed to the washer, is plotted against per cent total solids in the dilute black liquor return, two lines result. One is for digester operation, the second for washer operation. The point at which these two lines cross fixes the total solids content in the black liquor which is satisfactory to both the conditions of cooking and of washing. In this particular case, the total solids content is 0.229 or 22.9% and the dilute black liquor returns to the digester at 3.8% total solids.

If the washing conditions are changed, the value of black liquor solids which is mutually satisfactory to both the digester and washing system will also change. For example, consider the case where the four-press washing system shown in Fig. 2 is altered in operating conditions. In the illustration, all presses are fed at the same consistency, that of the pulp leaving the digester. How will operation be changed when presses 2, 3, and 4 are fed at 15% consistency instead of at the consistency leaving the digester? Discharge consistency from each press is still constant at 40%.

Basis of calculations: 43 lb. dry pulp (100 lb. dry wood fed to digester)

$$Y_2 = Y_3 = Y_4 = \frac{43}{0.15} - 43 = 286.6 - 43 = 243.6 \text{ lb.}$$

$$X_2 = X_3 = X_4 = 43$$

In the development of equations (14) and (15) for the press washing system $Y_1$ for press no. 1 did not enter into the calculations. Thus, these equations are still valid for calculating the relationship between $c_1$ and $c_2$ if the correct value of $Y$ is used.

Therefore,

$$\frac{Y}{1.5X} = \frac{243.6}{64.5} = 3.78$$

$$\left(\frac{Y}{1.5X} - 1\right)^3 = 21.4$$

$$\left(\frac{Y}{1.5X} - 0.5\right)^2 = 10.75$$

When $c_1 = 0.35$, from equation (15)

$$0.35 = (21.4 + 10.74 + 0.75)c_4$$
$$= 32.89c_4$$
$$c_4 = \frac{0.35}{32.89} = 0.01065$$

from equation (14)

$$c_2 = c_4(10.75 + 0.75)$$
$$= 0.01065 \times 11.50$$
$$= 0.1225$$

when $c_1 = 0.30$

$$c_2 = 0.30 \times \frac{11.50}{32.89} = 0.1050$$

when $c_1 = 0.26$

$$c_2 = 0.26 \times \frac{11.50}{32.89} = 0.0909$$

when $c_1 = 0.24$

$$c_2 = 0.24 \times \frac{11.50}{32.89} = 0.0839$$

when $c_1 = 0.22$

$$c_2 = 0.22 \times \frac{11.50}{32.89} = 0.0770$$

When these values of $c_2$ are plotted versus $c_1$ on the same chart as the values for black liquor solids in Table II, the black liquor solids which satisfy both the digester and the modified washing systems are 24.5% in the strong liquor and 9.0% in the dilute liquor return.

The black liquor concentration line just calculated applies to all conditions when digester yield is 43%. When digester yield is 48%,

$$Y = \frac{48}{0.15} - 48 = 320 - 48 = 320 - 48 = 272$$

$$\frac{Y}{1.5X} = \frac{272}{1.5 \times 48} = 3.78$$

completion of the calculation shows that when

$$c_1 = 0.20 \qquad c_2 = 0.0693$$
$$c_1 = 0.35 \qquad c_2 = 0.1220$$

which defines the line showing the relationship between feed and filtrate solids when the last three presses are fed at 15% consistency and digester yield is 48%.

*Vacuum Filter Washing.* Vacuum filters do not lend themselves to mathematical analysis as readily as do screw presses. The filtrate from each stage is usually split, part being used on the sprays of the preceding stage, the remaining larger portions being used to dilute the pulp entering the stage from which the filtrate is taken. This means that there are four different liquor concentrations involved in each stage: namely, concentrations of feed, sprays, filtrate, and pulp discharge. This multiplicity of variables makes mathematical analysis of the system very complicated.

For this work it is assumed that there will be a definite fixed ratio between total solids in the black liquor feed and in the filtrate from the first washer. This ratio will depend on the number of washers used in series in the system. A typical three-stage vacuum washer system is shown in Fig. 3. Here the per cent solids from the blow tank is 22.0, while the corresponding figure for the filtrate is 16.1%.

It will be assumed, therefore, that the total solids in the dilute liquor return will be equal to the fraction of total solids in the black liquor from the digester

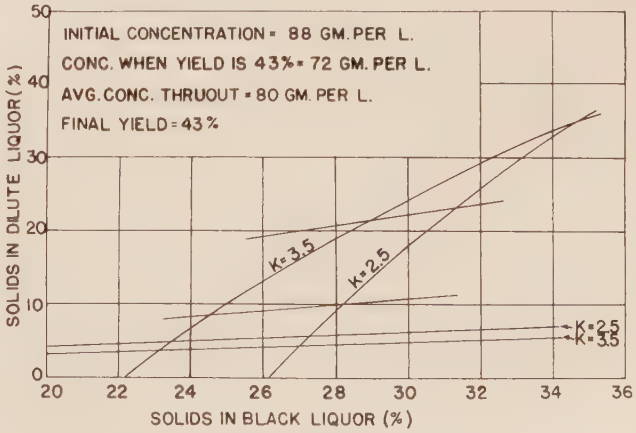


*Fig. 4. Washing and continuous cooking curves*

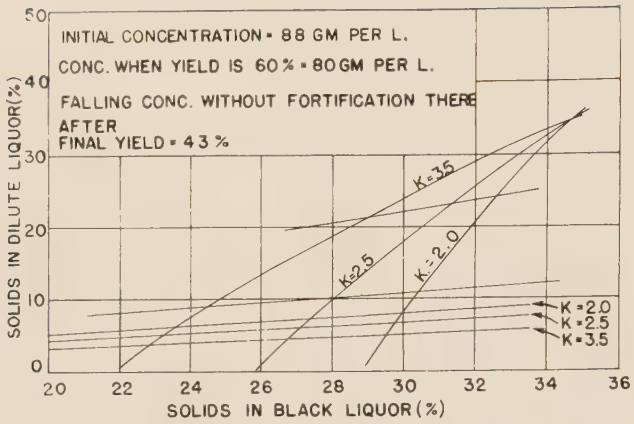


*Fig. 5. Washing and continuous cooking curves*

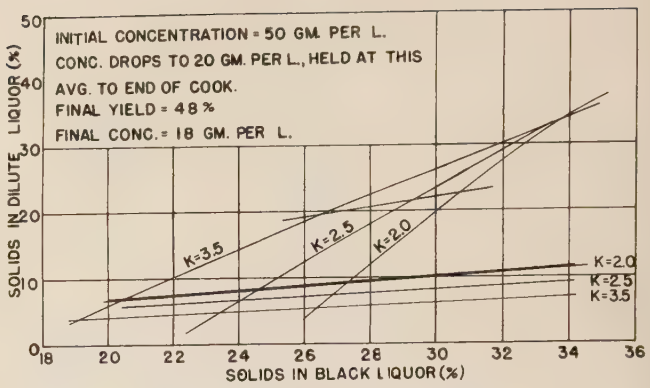


*Fig. 7. Washing and continuous cooking curves*

$\times 16.1/22.0$. It is admitted that this assumption is probably in error to a considerable degree but should suffice for estimating purposes. Using this ratio of 16.1/22.0, it is a simple matter to plot total solids of the strong black liquor feed versus total solids in the dilute filtrate. This line is independent of conditions used in the digester system. The intersection of this line with the total solids curve for the digester conditions will set the operating conditions for the digester and three-stage vacuum washers.

## CALCULATION OF EVAPORATION LOAD

In all cases where fortification liquor is used in the digesters, white liquor must be evaporated to increase its concentration above the usual strength of about 9.0% $Na_2O$ as it comes from the causticizing system. Therefore, two evaporation loads must be calculated. One of these consists of the evaporation necessary to increase the concentration of the black liquor solids going to the furnace. The second is the evaporation necessary to increase the strength of the white liquor from 9.0% to the arbitrarily set value of 32.0% $Na_2O$.

It is necessary to calculate the white liquor and black liquor quantities involved for each set of cooking conditions studied. Before this can be done, the quantities and concentrations of strong and weak black liquors must be evaluated for the various combinations of digester conditions and washing conditions. Therefore, a chart for each set of cooking conditions was made up. On each chart are plotted the lines defining the relationship between solid fractions in the strong and dilute liquors. The point of intersection between each digester line and the corresponding washer line defines the operating concentration.

Figure 4 represents the situation when cooking conditions are: initial concentration 88 grams per liter; average concentration, 80 grams per liter throughout; final concentration is 72 grams per liter when the final yield is 43%. Liquor ratios of 3.5 and 2.5 were used.

In Fig. 5 the cooking conditions are: initial concentration, 88 grams per liter; concentration held by fortification at an average of 80 grams per liter until 40% of the wood has dissolved; concentration at this point, 80 grams per liter; no further fortification as the cook proceeds to the final yield of 43%.

In Fig. 6 the cooking conditions are: initial concentration, 88 grams per liter; concentration held by fortification at an average of 80 grams per liter until 40% of the wood has dissolved; concentration at this point, 72 grams per liter; no further fortification as the cook proceeds to a final yield of 43%.

In Fig. 7 the cooking conditions are: initial concentration, 50 grams per liter; no fortification until concentration has dropped to 20 grams per liter; from this point to the final yield of 48%, concentration held by fortification at an average of 20 grams per liter; final concentration, 18 grams per liter.

In Fig. 8 the cooking conditions are: initial concentration, 22 grams per liter; concentration held by fortification at an average of 20 grams per liter throughout the cook to the final yield of 48%; final concentrations, 18 grams per liter.

The coordinates of the various points of intersection were read from each of the figures and are listed in Table VI.

A typical calculation of evaporation load is as follows:

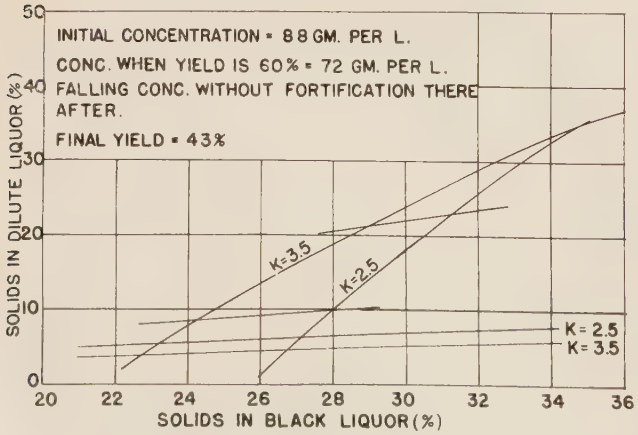
INITIAL CONCENTRATION = 88 GM. PER L.
CONC. WHEN YIELD IS 60% = 72 GM. PER L.
FALLING CONC. WITHOUT FORTIFICATION THEREAFTER.
FINAL YIELD = 43%
K=3.5
K=2.5
K=2.5
K=3.5
SOLIDS IN DILUTE LIQUOR (%)
SOLIDS IN BLACK LIQUOR (%)

*Fig. 6. Washing and continuous cooking curves*

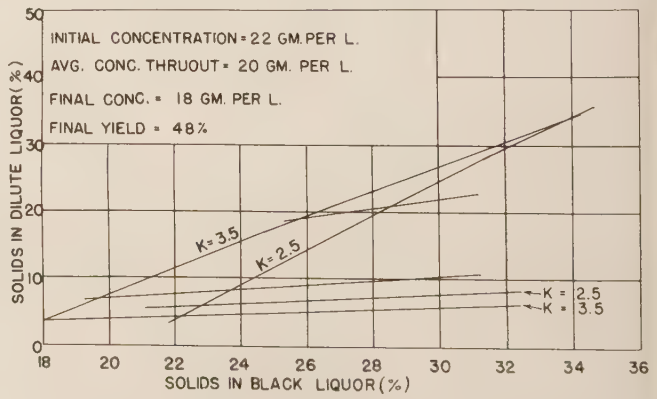
INITIAL CONCENTRATION = 22 GM. PER L.
AVG. CONC. THRUOUT = 20 GM. PER L.
FINAL CONC. = 18 GM. PER L.
FINAL YIELD = 48%
K=3.5
K=2.5
K=2.5
K=3.5
SOLIDS IN DILUTE LIQUOR (%)
SOLIDS IN BLACK LIQUOR (%)

*Fig. 8. Washing and continuous cooking curves*

**Table VI. Concentrations of Black Liquor to and from Washing System**

(Continuous pulping)

| Cooking conditions | Liquor ratio (K) | Four-stage press washing, all presses at digester consistency: Feed, % solids | Four-stage press washing, all presses at digester consistency: Filtrate, % solids | Four-stage press washing, last three stages at 15% consistency: Feed, % solids | Four-stage press washing, last three stages at 15% consistency: Filtrate, % solids | Three-stage vacuum washing: Feed, % solids | Three-stage vacuum washing: Filtrate, % solids |
|---|---|---|---|---|---|---|---|
| See Fig. 4 | 2.5 | 27.30 | 5.75 | 28.20 | 10.00 | 31.35 | 23.00 |
| | 3.5 | 23.10 | 3.70 | 24.50 | 8.50 | 28.97 | 21.30 |
| See Fig. 5 | 2.0 | 30.00 | 8.00 | 30.43 | 10.80 | 32.58 | 23.85 |
| | 2.5 | 27.10 | 6.00 | 28.05 | 10.2 | 31.30 | 22.90 |
| | 3.5 | 22.90 | 3.80 | 24.50 | 9.00 | 29.00 | 21.20 |
| | 2.5 | 27.17 | 6.40 | 28.00 | 9.90 | 31.33 | 23.00 |
| | 3.5 | 22.65 | 4.00 | 24.25 | 8.50 | 29.05 | 21.20 |
| See Fig. 7 | 2.0 | 27.25 | 9.30 | 27.30 | 9.50 | 30.70 | 22.70 |
| | 2.5 | 24.05 | 6.90 | 24.63 | 8.50 | 29.23 | 21.60 |
| | 3.5 | 19.10 | 4.00 | 20.60 | 7.20 | 26.45 | 19.70 |
| See Fig. 8 | 2.5 | 22.78 | 6.00 | 23.70 | 8.30 | 28.53 | 20.80 |
| | 3.5 | 17.95 | 3.50 | 19.73 | 6.90 | 25.65 | 18.80 |

Basis: 100 lb. dry wood
Cooking conditions:
Initial concentration = 88 grams $Na_2O$ per liter
Average concentration until 40% of wood removed = 80 grams per liter
Concentration at 40% wood removal = 80 grams per liter
No further fortification after 40% wood removal
Final yield = 43% of original wood
Liquor wood ratio = 2.0
Type of washing, four-stage screw press, consistency of feed to all presses the same as that leaving digester
Weight of black liquor from digester = $100k + A + P = 200 + 46.8 + 57 = 303.8$ lb.
Per cent total solids in black liquor (Table VI) = 30.0%

$$\frac{b}{a} \text{ (equation 11)} = \frac{200 \times 0.0791 + 0.32 \times 46.8 - 0.25 \times 57}{303.8 \times 0.300}$$

$$= \frac{15.82 + 14.96 - 14.25}{91.2} = \frac{16.54}{91.2}$$

$$= 0.1813$$

Total solids in dilute liquor, $a$ (Table VI) = 8.00%

$$b = 0.1813 \times 0.080 = 0.01455$$

$$y \text{ (equation 7)} = \frac{100 \times 0.32 \times 1.0 - 100 \times 2.0 \times 0.0791}{0.32 - 0.01455}$$

$$= \frac{16.18}{0.3054} = 53.1 \text{ lb.}$$

Black liquor from first press to evaporators = $303.8 - 1.5 \times 43 = 239.3$ lb.
Filtrate to evaporators = $239.3 - y = 239.3 - 53.1 = 186.2$ lb.
Total solids to evaporators = $239.3 \times 0.300 + 186.2 \times 0.080 = 71.9 + 14.9 = 86.8$ lb.
Weight of evaporated liquor (60% solids) = $86.8/0.60 = 145$ lb.
Evaporation load = $239.3 + 186.2 - 145 = 280.5$ lb.

Similar complete calculations for all conditions studied are listed in Table VII.

A sample calculation of the evaporation load required to concentrate the white liquor from 9.0 to 32% is shown below. The same conditions are used as were employed in calculating the black liquor evaporation load.

Total white liquor used = white liquor at digester entrance ($x$) + white liquor fortification ($A$) = $200 - 100 - 53.0 + 46.8 = 93.8$ lb.
Weight of $Na_2O = 93.8 \times 0.32 = 30.0$ lb.
White liquor from causticizer at 9% $Na_2O = 30/0.09 = 333$ lb.
White liquor evaporation = $333 - 94 = 239$ lb.
Total evaporation for the process = $239 + 281 = 520$ lb.

It will be noted that, in calculating the weight of $Na_2O$ from total white liquor used, the resulting value of $Na_2O$ is the per cent chemical used in the cook.

Table VIII contains the complete calculations for white liquor evaporation and the total evaporation load for all of the conditions studied.

## COMPARISON TO COMMERCIAL BATCH COOKING

The method of calculation developed above for continuous pulping can be applied to the standard batch type cook as practiced in most pulp mills. The same equations (5), (7), and (9) developed for fortification cooking, can be used here with the simplification that $A$, the weight of fortification liquor, is zero.

Equation (9) shows that there is an absolute minimum for liquor-wood ratio. This equation states, when $A = 0$, that:

$$\frac{b}{a} = \frac{100Kc - 0.25P}{\text{total solids in black liquor}}$$

For any conditions of yield and concentration, the value of the numerator decreases as $K$, the liquor-wood ratio, decreases. The limiting value for $K$ is that value which makes the numerator zero. Of course, the practical limit for $K$ is somewhat higher than this value because the actual yield will not decrease to the expected value unless there is sufficient excess chemical to carry the reaction to the desired yield.

For comparison purposes, the total evaporation requirements for a batch type cook will be calculated. It is assumed that cooking conditions are as listed below.

Initial concentration at digester entrance (including water in wood) = $c$ = 50 grams per liter = 4.77% $Na_2O$ (sp. gr. 1.05)
Concentration of white liquor ($d$) = 9.0% $Na_2O$
Liquor-wood ratio ($K$) = 3.5
Chip moisture = 50%
Yield of pulp = 48% of original dry wood
Basis of calculation: 100 lb. moisture-free wood
Total solids in black liquor (equation (5))

$$= \frac{100 \times 0.09\,(3.5 - 1) + 52 - y(0.09 - a)}{350 + 52}$$

$$= \frac{74.5 - y(0.09 - a)}{402}$$

from equation (7)

$$y = \frac{9 \times 2.5 - 350 \times 0.477}{0.09 - b} = \frac{5.8}{0.09 - b}$$

From equation (9)

$$\frac{b}{a} = \frac{16.7 - 0.25 \times 52}{402 \times \text{T.S.}} = \frac{3.7}{402 \times \text{T.S.}}$$

Assuming a value of 0.20 for fraction of total solids in the black liquor,

$$y(0.09 - a) = 74.5 - 402 \times 0.20$$

$$y = \frac{5.9}{a - 0.09}$$

## Table VII. Black Liquor Evaporation Load

(Continuous cooking)

| *Cooking conditions* | *Washing conditions* | *Liquor-wood ratio* (K) | *Weight black liquor, lb.* | *Total solids in black liquor* % | *Lb.* | b/a | a | b | *0.32*-b | y, *lb.* | *Black[a] liquor to evap., lb.* | *Filtrate[b] to evap., lb.* | *Total solids to evap., lb.* | *Total weight after evap., lb.* | *Black liquor evap., lb.* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| See Fig. 4 | Four-stage press, constant consistency | 2.5 | 364.7 | 27.30 | 99.6 | 0.2420 | 0.0575 | 0.0139 | 0.3061 | 91.2 | 300 | 209 | 93.9 | 156 | 353 |
| | | 3.5 | 459.7 | 23.10 | 106.3 | 0.2850 | 0.0370 | 0.0105 | 0.3095 | 169.0 | 395 | 226 | 99.8 | 167 | 452 |
| | Four-stage press, three presses at 15% consistency | 2.5 | 364.7 | 28.20 | 102.7 | 0.2340 | 0.1000 | 0.0234 | 0.2966 | 95.2 | 300 | 84 | 92.9 | 155 | 229 |
| | | 3.5 | 459.7 | 24.50 | 112.6 | 0.2690 | 0.0850 | 0.0228 | 0.2972 | 176.0 | 395 | 3 | 97.1 | 162 | 236 |
| | Three-stage vacuum filters | 2.5 | 364.7 | 31.35 | 114.3 | 0.2105 | 0.2300 | 0.0485 | 0.2715 | 104.0 | 394 | 0 | 91.5 | 152 | 242 |
| | | 3.5 | 459.7 | 28.97 | 133.4 | 0.2290 | 0.2130 | 0.0487 | 0.2713 | 193.0 | 435 | 0 | 92.5 | 154 | 281 |
| See Fig. 5 | Four-stage press, constant consistency | 2.0 | 303.8 | 30.00 | 91.2 | 0.1815 | 0.0800 | 0.0145 | 0.3055 | 53.0 | 239 | 186 | 86.6 | 144 | 281 |
| | | 2.5 | 352.3 | 27.12 | 95.5 | 0.2093 | 0.0600 | 0.0125 | 0.3075 | 91.8 | 288 | 196 | 89.7 | 150 | 334 |
| | | 3.5 | 449.6 | 22.90 | 103.0 | 0.2630 | 0.0380 | 0.0100 | 0.3100 | 168.5 | 385 | 217 | 96.5 | 161 | 441 |
| | Four-stage press, three presses at 15% consistency | 2.0 | 303.8 | 30.43 | 92.5 | 0.1790 | 0.1080 | 0.0193 | 0.3007 | 52.7 | 239 | 127 | 86.0 | 143 | 223 |
| | | 2.5 | 352.3 | 28.05 | 98.8 | 0.2030 | 0.1020 | 0.0207 | 0.2993 | 94.4 | 288 | 85 | 89.5 | 149 | 224 |
| | | 3.5 | 449.6 | 24.50 | 110.3 | 0.2453 | 0.0900 | 0.0220 | 0.2980 | 175.3 | 385 | 4 | 94.7 | 158 | 231 |
| | Three-stage vacuum filters | 2.0 | 303.8 | 32.58 | 99 0 | 0.1673 | 0.2380 | 0.0398 | 0.2802 | 57.8 | 357 | 0 | 85.2 | 142 | 215 |
| | | 2.5 | 352.3 | 31.30 | 110.0 | 0.1820 | 0.2290 | 0.0417 | 0.2783 | 101.5 | 379 | 0 | 86.7 | 144 | 235 |
| | | 3.5 | 449.6 | 29.00 | 130.5 | 0.2070 | 0.2120 | 0.0440 | 0.2760 | 189.5 | 425 | 0 | 90.2 | 150 | 275 |
| See Fig. 6 | Four-stage press, constant consistency | 2.5 | 343.6 | 27.17 | 93.5 | 0.1897 | 0.0640 | 0.01214 | 0.3079 | 91.6 | 279 | 188 | 87.8 | 146 | 321 |
| | | 3.5 | 438.6 | 22.65 | 99.4 | 0.2370 | 0.0480 | 0.00948 | 0.3105 | 168.5 | 374 | 206 | 94.3 | 157 | 423 |
| | Four-stage press, three presses at 15% consistency | 2.5 | 343.6 | 28.00 | 96.4 | 0.1837 | 0.0990 | 0.01818 | 0.3018 | 93.5 | 279 | 86 | 85.8 | 143 | 223 |
| | | 3.5 | 438.6 | 24.25 | 106.4 | 0.2210 | 0.0850 | 0.01880 | 0.3012 | 173.7 | 374 | 6 | 91.3 | 152 | 228 |
| | Three-stage vacuum filters | 2.5 | 343.6 | 31.33 | 107.7 | 0.1645 | 0.2300 | 0.03780 | 0.2822 | 100.0 | 370 | 0 | 84.8 | 141 | 229 |
| | | 3.5 | 438.6 | 29.05 | 127.5 | 0.1846 | 0.2120 | 0.03920 | 0.2808 | 186.3 | 414 | 0 | 88.0 | 147 | 267 |
| See Fig. 7 | Four-stage press, constant consistency | 2.0 | 278.5 | 27.25 | 75.8 | 0.0610 | 0.0930 | 0.0057 | 0.3143 | 72.5 | 206 | 134 | 68.4 | 114 | 226 |
| | | 2.5 | 323.7 | 24.05 | 77.8 | 0.0690 | 0.0690 | 0.0048 | 0.3152 | 115.5 | 251 | 136 | 70.6 | 118 | 269 |
| | | 3.5 | 414.1 | 19.10 | 79.1 | 0.0869 | 0.0400 | 0.0035 | 0.3165 | 201.0 | 342 | 140 | 70.9 | 118 | 364 |
| | Four-stage press, three presses at 15% consistency | 2.0 | 278.5 | 27.30 | 76.0 | 0.0608 | 0.0950 | 0.0058 | 0.3142 | 72.5 | 206 | 128 | 68.4 | 114 | 220 |
| | | 2.5 | 323.7 | 24.63 | 78.5 | 0.0683 | 0.0850 | 0.0058 | 0.3142 | 116.5 | 251 | 84 | 69.0 | 115 | 220 |
| | | 3.5 | 414.1 | 20.60 | 85.3 | 0.0807 | 0.0720 | 0.0058 | 0.3142 | 203.0 | 341 | −3 | 69.7 | 115 | 223 |
| | Three-stage vacuum filter | 2.0 | 278.5 | 30.70 | 83.7 | 0.0552 | 0.2270 | 0.0122 | 0.3078 | 73.9 | 306 | 0 | 67.4 | 112 | 194 |
| | | 2.5 | 323.7 | 29.23 | 94.5 | 0.0568 | 0.2160 | 0.0123 | 0.3077 | 118.3 | 334 | 0 | 71.5 | 119 | 215 |
| | | 3.5 | 414.1 | 26.45 | 109.3 | 0.0628 | 0.1970 | 0.0124 | 0.3076 | 207.0 | 338 | 0 | 67.7 | 113 | 225 |
| See Fig. 8 | Four-stage press, consistency | 2 5 | 344.8 | 22.78 | 78.7 | 0.0748 | 0.0600 | 0.0045 | 0.3155 | 135.5 | 273 | 137 | 70.4 | 117 | 293 |
| | | 3.5 | 443.6 | 17.95 | 79.8 | 0.0953 | 0.0350 | 0.0033 | 0.3167 | 230.0 | 372 | 142 | 71.7 | 120 | 394 |
| | Four-stage press, three presses at 15% consistency | 2.5 | 344.8 | 23.70 | 81.8 | 0.0718 | 0.0830 | 0.0060 | 0.3140 | 136.3 | 273 | 64 | 70.0 | 117 | 219 |
| | | 3.5 | 443.6 | 19.73 | 87.6 | 0.0867 | 0.0690 | 0.0060 | 0.3140 | 231.0 | 372 | −31 | 71.1 | 119 | 220 |
| | Three-stage vacuum filter | 2.5 | 344.8 | 28.53 | 98.4 | 0.0598 | 0.2080 | 0.0124 | 0.3076 | 139.3 | 333 | 0 | 69.5 | 116 | 217 |
| | | 3.5 | 443.6 | 25.65 | 113.8 | 0.0668 | 0.1880 | 0.0126 | 0.3074 | 236.5 | 380 | 0 | 71.5 | 119 | 261 |

[a] For four-stage presses with consistency of feed constant, black liquor to evaporation = total B.L. − 1.5 yield. For four-stage presses with 15% consistency feed to three presses, black liquor to evaporation = total B.L. − 1.5 yield. For three-stage vacuum filters, black liquor to evaporation = total B.L. $\times \frac{22.0}{16.1} - y$.

[b] For four-stage presses with consistency of feed constant, filtrate to evaporator = B.L. to evaporator − $y$. For four-stage presses with 15% consistency feed to three presses, filtrate to evaporator = 272 − 1.5 yield − $y$. For three-stage vacuum filters, filtrate to evaporator = 0.

**Table VIII. White Liquor and Total Evaporation Loads**

(Continuous cooking)

| Cooking conditions | Washing conditions | Liquor-wood ratio (K) | White liquor to digester entrance (X), lb. | Fortification liquor, (A), lb. | Total white liquor, lb. | Weight of $Na_2O$, lb. | Weight of 9% white liquor, lb. | White liquor evap., lb. | Black liquor evap., lb. | Total evap., lb. |
|---|---|---|---|---|---|---|---|---|---|---|
| See Fig. 3 | Four-stage press, constant consistency | 2.5 | 58.8 | 57.9 | 116.7 | 37.3 | 412 | 295 | 353 | 648 |
| | | 3.5 | 81.0 | 52.7 | 133.7 | 42.8 | 475 | 341 | 452 | 793 |
| | Four-stage press, three presses at 15% consistency | 2.5 | 54.8 | 57.9 | 112.7 | 36.1 | 401 | 288 | 229 | 517 |
| | | 3.5 | 74.0 | 52.7 | 126.7 | 40.5 | 450 | 323 | 236 | 559 |
| | Three-stage vacuum filters | 2.5 | 46.0 | 57.9 | 103.9 | 33.3 | 370 | 266 | 242 | 508 |
| | | 3.5 | 57.0 | 52.7 | 109.7 | 35.2 | 391 | 281 | 281 | 562 |
| See Fig. 4 | Four-stage press, constant consistency | 2.0 | 47.0 | 46.8 | 93.8 | 30.0 | 334 | 240 | 281 | 521 |
| | | 2.5 | 58.2 | 45.3 | 103.5 | 33.1 | 368 | 264 | 334 | 598 |
| | | 3.5 | 81.5 | 42.6 | 124.1 | 39.7 | 441 | 317 | 441 | 758 |
| | Four-stage press, three presses at 15% consistency | 2.0 | 47.3 | 46.8 | 94.1 | 30.1 | 334 | 240 | 223 | 463 |
| | | 2.5 | 55.5 | 45.3 | 100.8 | 32.3 | 359 | 258 | 224 | 482 |
| | | 3.5 | 74.7 | 42.6 | 117.3 | 37.5 | 417 | 300 | 231 | 531 |
| | Three-stage vacuum filters | 2.0 | 42.2 | 46.8 | 89.0 | 28.5 | 317 | 228 | 215 | 443 |
| | | 2.5 | 48.5 | 45.3 | 93.8 | 30.0 | 333 | 239 | 235 | 474 |
| | | 3.5 | 60.5 | 42.6 | 103.1 | 33.0 | 366 | 263 | 275 | 538 |
| See Fig. 5 | Four-stage press, constant consistency | 2.5 | 58.4 | 36.6 | 95.0 | 30.4 | 338 | 243 | 321 | 564 |
| | | 3.5 | 81.5 | 31.6 | 113.1 | 36.2 | 402 | 289 | 423 | 712 |
| | Four-stage press, three stages at 15% consistency | 2.5 | 56.5 | 36.6 | 93.1 | 29.8 | 331 | 238 | 223 | 461 |
| | | 3.5 | 76.3 | 31.6 | 107.9 | 34.5 | 383 | 275 | 228 | 503 |
| | Three-stage vacuum filters | 2.5 | 50.0 | 36.6 | 86.6 | 27.7 | 308 | 221 | 229 | 450 |
| | | 3.5 | 63.7 | 31.6 | 95.3 | 30.5 | 339 | 244 | 267 | 511 |
| See Fig. 6 | Four-stage press, constant consistency | 2.0 | 27.5 | 26.5 | 54.0 | 17.8 | 198 | 144 | 226 | 370 |
| | | 2.5 | 34.5 | 21.7 | 56.2 | 18.0 | 200 | 144 | 269 | 413 |
| | | 3.5 | 49.0 | 12.1 | 61.1 | 19.6 | 218 | 157 | 364 | 521 |
| | Four-stage press, three stages at 15% consistency | 2.0 | 27.5 | 26.5 | 54.0 | 17.3 | 191 | 137 | 220 | 357 |
| | | 2.5 | 33.5 | 21.7 | 55.2 | 17.7 | 197 | 142 | 220 | 362 |
| | | 3.5 | 47.0 | 12.1 | 59.1 | 18.9 | 210 | 151 | 223 | 374 |
| | Three-stage vacuum filters | 2.0 | 26.1 | 26.5 | 52.6 | 16.8 | 187 | 134 | 194 | 328 |
| | | 2.5 | 31.7 | 21.7 | 53.4 | 17.1 | 190 | 137 | 215 | 352 |
| | | 3.5 | 43.0 | 12.1 | 55.1 | 17.6 | 196 | 141 | 225 | 366 |
| See Fig. 7 | Four-stage press, constant consistency | 2.5 | 14.5 | 42.8 | 57.3 | 18.3 | 204 | 147 | 293 | 440 |
| | | 3.5 | 20.0 | 41.6 | 61.6 | 19.7 | 219 | 157 | 394 | 549 |
| | Four-stage press, three presses at 15% consistency | 2.5 | 13.7 | 42.8 | 56.5 | 18.1 | 201 | 145 | 219 | 364 |
| | | 3.5 | 19.0 | 41.6 | 60.6 | 19.4 | 216 | 155 | 220 | 375 |
| | Three-stage vacuum filters | 2.5 | 10.7 | 42.8 | 53.5 | 17.1 | 190 | 136 | 217 | 353 |
| | | 3.5 | 13.5 | 41.6 | 55.1 | 17.6 | 196 | 141 | 261 | 402 |

$$\frac{b}{a} = \frac{3.7}{80.4} = 0.460$$

$$\frac{5.9}{a - 0.09} = \frac{5.8}{0.09 - 0.0460\,a}$$

$$a\,(5.8 + 5.9 \times 0.0460) = 0.32\,(5.8 + 5.9)$$

$$a = \frac{1.053}{6.071} = 0.1738$$

$$b = 0.174 \times 0.0460 = 0.0080$$

$$y = \frac{5.9}{0.0838} = 70.8$$

The values of $a$, the concentration of $Na_2O$ in the dilute black liquor returned to the digester, were calculated for assumed values of total solids in the black liquor going to the blow tank. The calculated values are listed in Table IX for $K$ values of 3.0 and 3.5.

The same washing systems were considered for washing the pulp for the batch digester as were studied for continuous pulping. The same equations hold in this case and the calculations were carried out in the same manner. The results of these calculations are listed in Table X. Only two values were calculated for each washing system since the relationship between total solids in the feed and filtrate dissolved solids is represented by a straight line.

The data in Tables IX and X were plotted in a manner similar to that used for continuous pulping in order to determine, by the intersection of the digester curve with the various washing lines, the correct black liquor solids for the operating condition. The graphical representation is shown in Fig. 9. The correct points of intersection were read from Fig. 9 and are listed in Table XI.

Calculation of the evaporation load required for batch pulping is simplified somewhat because there is no white liquor to be evaporated. The white liquor is used as it comes from the causticizer at 9% $Na_2O$.

**Table IX. Total Solids in Strong and Weak Black Liquor**

(Batch cooking)

| Total solids in strong black liquor | Total solids in weak black liquor (a) K = 3.5 | K = 3.0 |
|---|---|---|
| 25 | 0.425 | ... |
| 21 | ... | 0.195 |
| 20 | 0.174 | 0.099 |
| 19 | ... | 0.016 |
| 18 | 0.055 | −0.084 |
| 17 | −0.051 | ... |

**Table X. Relationship Between Dissolved Solids in Black Liquor Feed and Filtrate in the Washing System**

(Batch cooking)

| Liquor ratio K | Total solids in feed ($c_1$) | Total solids in filtrate ($c_2$): Four press washers, all presses at same consistency | Total solids in filtrate ($c_2$): Four press washers, last three presses fed at 15% consistency | Total solids in filtrate ($c_2$): Three-stage vacuum filters |
|---|---|---|---|---|
| 3.5 | 0.10 | 0.0205 | 0.0350 | 0.0732 |
| | 0.20 | 0.0412 | 0.0700 | 0.1464 |
| 3.0 | 0.10 | 0.0252 | 0.0350 | 0.0732 |
| | 0.20 | 0.0504 | 0.0700 | 0.1462 |

**Table XI. Concentration of Black Liquor to and from Washing System**
(Batch cooking)

| Liquor ratio | Four-stage press washing, all presses at digester consistency: Feed, % solids | Four-stage press washing, all presses at digester consistency: Filtrate, % solids | Four-stage press washing, last three presses at 15% consistency: Feed, % solids | Four-stage press washing, last three presses at 15% consistency: Filtrate, % solids | Three-stage vacuum washing: Feed, % solids | Three-stage vacuum washing: Filtrate, % solids |
|---|---|---|---|---|---|---|
| 3.5 | 17.7 | 3.5 | 18.2 | 6.2 | 19.5 | 14.2 |
| 3.0 | 19.4 | 5.0 | 19.6 | 6.7 | 20.6 | 15.0 |

The calculations for black liquor evaporation are similar to those illustrated in continuous pulping and are listed in Table XII.

## DISCUSSION OF RESULTS

A comparison of evaporation load required by continuous versus batch pulping may be made by a study of Tables VIII and XII. It can be seen that continuous pulping at high concentration (80 grams $Na_2O$) for the production of dissolving grades of pulp will require about 41% more evaporation than batch cooking at a liquor-wood ratio of 3.0. If the batch process is carried out at a liquor-wood ratio of 3.5, the continuous method requires 32% more evaporation. This latter figure of

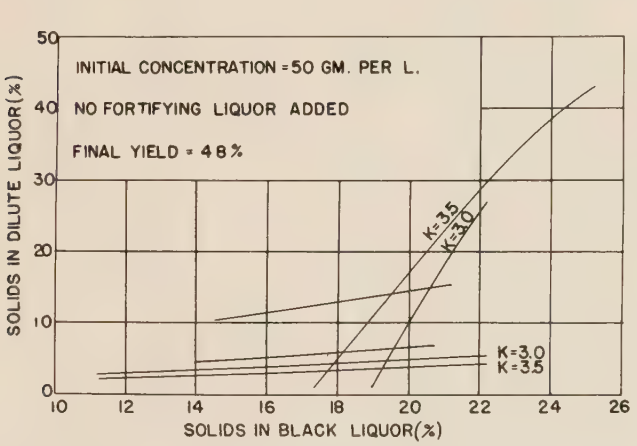


*Fig. 9. Washing and batch cooking curves*

32% is a more reasonable comparison because it seems improbable that a batch cook can be carried out at a liquor ratio of 3.0 and a starting concentration of 50 grams per liter unless wood moisture is considerably less than 50%. Under the conditions specified, only 14.3% $Na_2O$ would be used, which seems too low for satisfactory pulping.

High concentration pulping will use much more chemical in the process than batch pulping. The increase in chemical will be $[(30.1 - 16.4)/16.4] \times 100 = 83.5\%$. This increased usage should be considered an inventory rather than a direct cost since most of the chemical is recovered. However, recovery costs will increase because of the increased weight of solids to be handled and losses due to handling will increase.

Chemical losses in the washed pulp are much higher for the continuous process. The concentration of total solids in the liquid leaving with the washed pulp is 0.91% as compared to 0.54% for the batch process. This involves, per ton of moisture-free pulp, a loss of:

For continuous methods, $\frac{0.0091 \times 1.5 \times 43 \times 2000}{43} =$ 27.4 lb. T.S./ton pulp

For batch methods, $\frac{0.0054 \times 1.5 \times 48 \times 2000}{48} =$ 16.2 lb. T.S./ton pulp

The active chemical loss is greater in proportion for continuous pulping because of the higher content of alkali in the black liquor solids. The active alkali loss, expressed as pounds of $Na_2SO_4$ per ton of dry pulp is:

For continuous pulping, $27.4 \times 0.179 \times \frac{142}{62} = 11.45$ lb.

For batch pulping, $16.2 \times 0.020 \times \frac{142}{62} = 0.75$ lb.

The very low figure for batch pulping is caused by the combination of four stages of washing instead of the usual three stages and the fact that the washed pulp carries very little liquid with it because of its 40% consistency. The high loss accompanying the continuous process is accounted for, primarily, by the much higher ratio of active alkali to total solids in the black liquor. For continuous pulping at high concentration this ratio is 0.179 as compared to 0.020 for batch pulping. It might be advisable, in view of the high theoretical losses in washed pulp, to use one additional stage of washing for high concentration continuous pulping. This should make the washing losses comparable for the two processes.

It would appear, therefore, that high concentration continuous pulping will be more costly than batch pulping. It must be borne in mind, however, that the more costly process produces a dissolving grade of pulp

**Table XII. Black Liquor Evaporation Load**
(Batch Cooking)

| Washing conditions | Liquor-wood ratio (K) | Weight black liquor, lb. | Total solids in black liquor % | Total solids in black liquor lb. | b/a | a | b | 0.09 − b | y, lb. | Black[a] liquor to evap., lb. | Filtrate[b] to evap., lb. | Total solids to evap., lb. | Black liquor evap., lb. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Four-stage press, constant consistency | 3.0 | 352 | 19.4 | 68.4 | 0.0197 | 0.050 | 0.00099 | 0.0890 | 41.0 | 280 | 239 | 66.2 | 409 |
| | 3.5 | 402 | 17.7 | 71.3 | 0.0519 | 0.035 | 0.00182 | 0.0882 | 65.8 | 333 | 264 | 67.7 | 481 |
| Four-stage press, three presses at 15% consistency | 3.0 | 352 | 19.6 | 69.0 | 0.0196 | 0.067 | 0.00131 | 0.0887 | 41.2 | 280 | 159 | 66.6 | 328 |
| | 3.5 | 402 | 18.2 | 73.0 | 0.0507 | 0.062 | 0.00314 | 0.0869 | 66.8 | 330 | 133 | 68.2 | 349 |
| Three-stage vacuum filters | 3.0 | 352 | 20.6 | 72.5 | 0.0186 | 0.150 | 0.00280 | 0.0872 | 41.8 | 439 | 0 | 66.1 | 329 |
| | 3.5 | 402 | 19.5 | 78.5 | 0.0472 | 0.142 | 0.00670 | 0.0833 | 69.7 | 480 | 0 | 68.5 | 366 |

[a] For four-stage presses with consistency of feed constant, black liquor to evaporator = total black liquor − 1.5 yield. For four-stage presses with 15% consistency feed to three presses, black liquor to evaporator = total black liquor − 1.5 yield. For three-stage vacuum filters, black liquor to evaporator = total black liquor $\times \frac{22.0}{16.1} - y$.

[b] For four-stage presses with consistency of feed constant, filtrate to evaporation = B.L. to evaporator − $y$. For four-stage presses with 15% consistency feed to three presses, filtrate to evaporator = 272 − 1.5 yield − $y$. For three-stage vacuum filters, filtrate to evaporator = 0.

as compared to the product of batch pulping, which is a papermaking grade. If the two processes were to be used for producing a dissolving grade of pulp, the increased pulping cost of the continuous method would be counterbalanced by the elimination of prehydrolysis and the increase in yield. The yield for dissolving grades made continuously is about 43% as compared to 35 to 38 per cent for prehydrolyzed pulps.

A much fairer comparison between continuous and batch methods is obtained when the comparison is made on processes which produce the same grade of pulp. Such a comparison is available in the case of the 50 grams $Na_2O$ per liter continuous cook versus the batch cook. The liquor-wood ratio for the continuous cook is 2.0; for the batch cook, 3.5. This low ratio for continuous cooking is permissible for two reasons. Active alkali is kept in great excess because of fortification, even though the liquor ratio is low. Secondly, a continuous digester permits the retention of a large amount of liquor in the digester which amounts to a very high liquor-wood ratio during cooking, even though the ratio of pounds of liquid added per pound of wood is low.

In the comparison, using four-stage screw presses with 15% consistency feed to the last three presses, the values are as follows. The continuous method requires 357 lb. of evaporation per 100 lb. of wood while the comparable evaporation for the batch method is 349. In other words, the evaporation costs of the two processes can be considered as being equal.

Chemical requirements are about the same in both cases. The continuous process uses 17.8% chemical while batch pulping requires 16.6% $Na_2O$. The increased chemical costs for continuous pulping over batch methods should, therefore, be considered as negligible.

Chemical losses in washing are much higher for the continuous process. In the first place, the concentrations of total solids in the black liquor are much higher because of the greater recirculation of black liquor through the digester and the introduction of less water in the white liquor. This accounts for an increase in the total dissolved solids leaving with the washed pulp, amounting to an increase of $[(25.1 - 16.2)/16.2] \times 100 = 55\%$ in the total solids leaving in the pulp. The per cent loss in active alkali, however, is higher because the total solids from continuous pulping contain 6.1% active alkali as compared to 2.0% for batch methods. The $Na_2SO_4$ losses per ton in these two cases are:

$$\text{For continuous pulping, } 25.1 \times 0.061 \times \frac{142}{62} = 3.55 \text{ lb.}$$

$$\text{For batch pulping, } 16.2 \times 0.020 \times \frac{142}{62} = 0.75 \text{ lb.}$$

In other words, the active alkali loss in the washed pulp from continuous pulping is almost five times that of batch pulping. Since both losses are numerically small it seems quite doubtful that an additional stage of washing would be warranted.

The evaporation and chemical consumption data in Table VIII show that, in high concentration continuous pulping, it is advisable to terminate fortification of the cooking liquor as early in the cook as possible. When the cooking liquor is held at an average of 80 grams $Na_2O$ until the final yield of 43% is reached, using a liquor-wood ratio of 2.5, the evaporation load is 517 lb. of water per 100 lb. of dry wood. When fortification of liquor is terminated so that the concentration is 80 grams per liter when 40% of the wood and the concentration decreases thereafter, the evaporation load decreases to 483 lb., a decrease of 6.6%. A further decrease of 4.2% in evaporation load can be realized if the fortification liquor is cut back so that concentration is 72 g.p.m. when wood removal is 40%. These evaporation loads were calculated for a four-stage screw press washing system, using 15% consistency feed to the last three presses.

A similar reduction in per cent chemical used during the cook occurs with a cutback in fortifying chemical. When fortification is reduced so that concentration falls after the point of 40% wood removal at a concentration of 80 grams per liter, chemical usage reduces from 36.1 to 32.3% of the original wood. A further reduction to 29.8 occurs when the cutback is such that the concentration drop begins at 40% wood removal and a concentration of 72 grams per liter.

It can, therefore, be seen that definite savings in both evaporation and chemical usage can be realized from a reduction in the amount of fortification. Further experimental work is needed to determine how far the cook must have progressed before concentration can be allowed to fall and still produce a pulp of low pentosan content.

In low concentration pulping, it was thought that, by increasing the initial concentration from 20 to 50 grams per liter, the consequent reduction in fortifying chemical might result in a saving in evaporation or chemical usage. Table VIII shows that no such saving can be realized. The only advantage that might be realized by increased initial concentration is the increased reaction rate which might result.

## CONCLUSIONS

High concentration continuous pulping for the production of dissolving pulps will be more costly than batch pulping for papermaking grades of pulp. The continuous method requires 32% more evaporation and uses 83.5% more chemical than batch methods. An additional stage of washing is necessary in continuous pulping to make the washing losses comparable to those for batch pulping.

The increased costs of washing, evaporation, and processing of alkali in this process are counterbalanced by savings realized from the elimination of prehydrolyzing before pulping and by a substantial decrease in wood cost.

Low concentration continuous pulping for the production of papermaking grades of pulp is as efficient in evaporation and chemical usage as batch methods in use at the present time. Alkali losses in the washed pulp are somewhat higher but do not warrant the use of an additional stage of washing.

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# A Study on the Effects of Instrumental Variables on 75° Specular Gloss

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**The work presented in this report was initiated for the purpose of determining the effects of instrumental variables on specular gloss measured in accordance with the TAPPI Tentative Standard T 480 m-51. The work resulted as a consequence of difficulties experienced in standardizing several instruments based on the TAPPI Standard. The investigation covers the effects of the following instrumental variables on the gloss: (1) the incident angle, (2) the receiving angle in the plane of measurement, (3) the receiving angle in the plane perpendicular to that of measurement, (4) the diameter of the receptor aperture, (5) the position of the image of the source, (6) the size of the image of the source, (7) the distance from the test area to the incident lens, (8) the size of the source aperture, and (9) the spectral characteristics. The results of this investigation indicate the desirability of reducing the tolerances below those listed in the TAPPI Standard. Suggested tolerances are presented in Table III of this report.**

The results of instrumental measurements of specular gloss depend very strongly on the geometric and optical properties of the instrument used for the measurements. To assure a common gloss scale throughout the paper industry, TAPPI has set up a Tentative Standard T 480 m-51 covering the requirements for a 75° glossmeter. Since the publication of the TAPPI method, we have had occasion to standardize several instruments against ceramic tiles furnished and calibrated by the National Bureau of Standards. During this work difficulties were experienced in that we failed to obtain satisfactory agreement between instruments and, moreover, we failed to obtain instrumental readings which agreed with the values assigned to the tiles. Discrepancies of as much as 8.5 gloss units were observed. In view of this situation, it was believed desirable to study the instrumental variables of the 75° glossmeter. This study has been undertaken to investigate the effects on the measured gloss of variations in (1) the incident angle, (2) the receiving angle in the plane of measurement, (3) the receiving angle in the plane perpendicular to that of measurement, (4) the diameter of the receptor aperture, (5) the position of the image of the source, (6) the size of the image of the source, (7) the distance from the test area to the incident lens, (8) the size of the source aperture, and (9) the spectral characteristics of the source and receptor. Hammond and Nimeroff have made a similar study on a 60° glossmeter (*1*). A description of the instrument used in this study together with the results and a list of suggested instrumental tolerances are presented in the following sections of this report.

## DESCRIPTION OF THE INSTRUMENT

A schematic diagram of the instrument used throughout the study is shown in Fig. 1. The illuminating system consists of a straight spiral filament lamp, A, followed by a pair of simple biconvex lenses of suitable focal length, B, and a source diaphragm, C. This system is mounted in a machined casting, and is attached to plate, H. The incident beam of light impinges on the sample through a slot of adequate size in the plate, H. The sample is maintained flat by means of a special holder, J, the details of which are shown in Fig. 2. A vacuum applied to the holder serves to hold the sample in intimate contact with the bronze plate.

The light reflected from the sample enters the diffusing sphere, K, through a receptor aperture, E. The interior wall of the sphere is coated with magnesium oxide. The coating was considered to be adequately uniform when the measured intensities of reflected light from various sections of the wall did not differ by more than $\pm 0.5\%$.

The intensity of the light entering the sphere, K, is measured with a barrier layer type photocell (Weston type 594 YR) fitted with a Viscor filter. The photocell is mounted with its sensitive surface flush with the wall of the sphere and at a position 90° from the aperture, E. Two baffles, G, are mounted near the photocell to prevent the direct rays from the aperture, E, and the first reflected rays from the sphere wall from reaching the photocell. The photocurrent is measured with a Rubicon galvanometer having a coil resistance of 445 ohms, a period of 4.9 sec., a sensitivity of 0.0015 microamp. per mm. of deflection and a scale length of 100 mm. In this study we are concerned only with small variations, therefore small nonlinearities of the photocell-galvanometer circuit have an insignificant influence on the results.

## PROCEDURE

Before starting the study on instrumental variables, an experiment was made to determine the effects of handling the specimens on the observed readings. This experiment involved 30 samples of paper, each of which was tested on each of 3 successive days. An analysis of variance of the results indicated that with normal handling the same identical specimens could be safely used throughout the study.

For the studies on the instrumental variables, eight "white" paper samples selected for maximum uniformity of gloss in the test area and four tiles evaluated by the National Bureau of Standards were used. A description of these samples is given in Table I. The paper specimens were mounted in chipboard frames to facilitate careful handling.

For the study of a given variable, each of the 12 samples was measured in an order determined from a

L. W. Zabel, Physicist, Kimberly-Clark Corp., Neenah, Wis., and W. A. Wink, Technical Associate, The Institute of Paper Chemistry, Appleton, Wis.

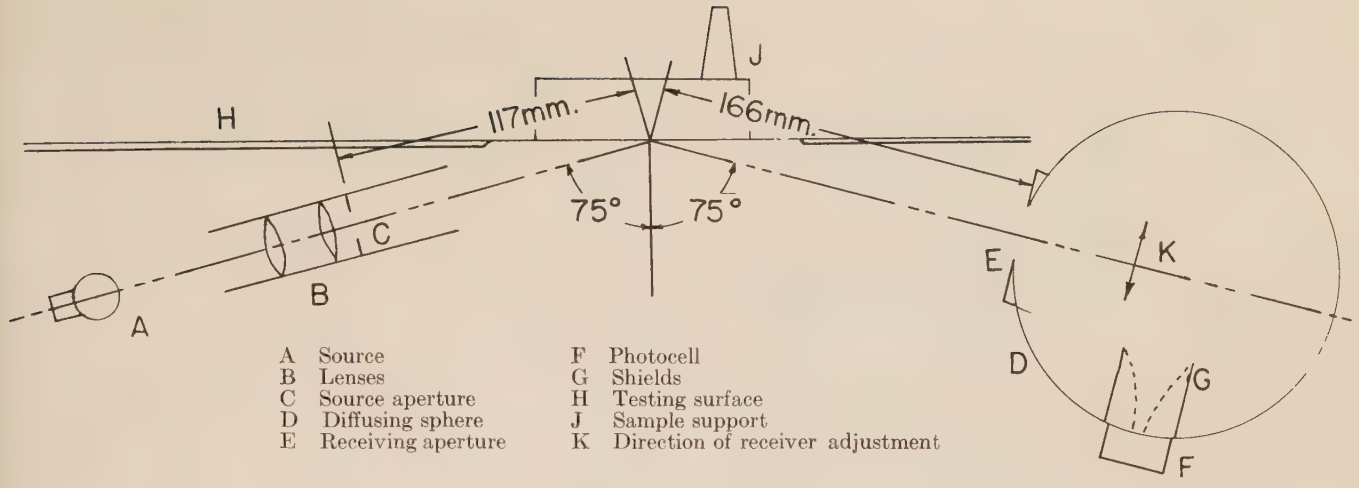



*Fig. 1. Apparatus*

table of random numbers. This was then repeated the desired number of times, each time using a different set of random numbers. This procedure was employed to minimize a dependence of the results on the order of sample measurement.

The standardization of the instrument was accomplished in each case by careful adjustment of the zero and full scale readings on the galvanometer. The zero was adjusted with a black cavity over the sample opening. A piece of polished black glass (index of refraction = 1.54) served as a standard for the full scale adjustment.

Goniophotometric curves were obtained on all samples to assist in the interpretation of results obtained in studying the instrumental variables of the 75° glossmeter (*2*).

### *The Incident Angle*

With all of the other variables held constant, the incident angle was varied in three steps and gloss measurements were made on each of the 12 specimens. These results are represented in Fig. 3 where the instrumental readings are plotted as a function of the incident angle. The slopes of these curves are a direct measurement of the sensitivity of the gloss readings to changes in incident angle. The slopes for the tiles are as high or higher than those for papers at the same gloss levels. The maximum slope for any single sample is 2.5 gloss units per degree. A high slope for the tiles is a desirable feature if they are to serve as gloss standards. For the study of this variable, ten replications per sample were used. An analysis of variance of the resulting data indicated that four replications would result in adequate sensitivity for the subsequent work.

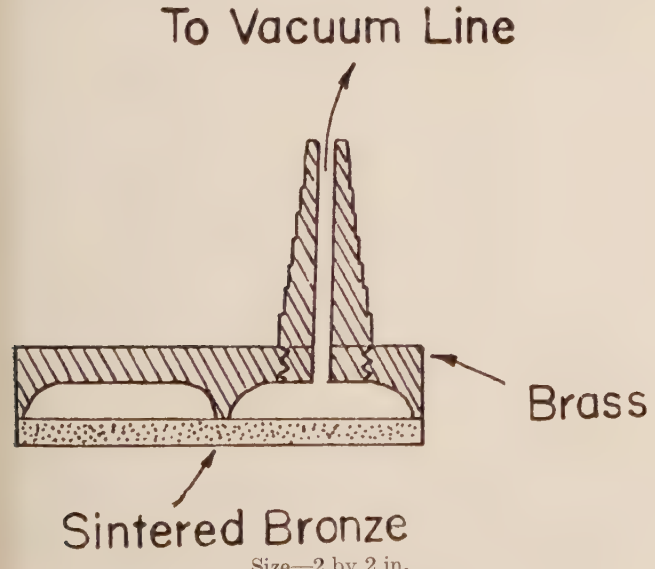



*Fig. 2. Sample holder*

### *The Receiving Angle in the Plane of Measurement*

The angle of incidence was held constant and the position of the receptor moved to vary the receiving angle. The receiving angle is defined as the angle between the normal to the plane of the sample and a line connecting the center of the test area and the center of the receptor aperture. Figure 4 is a plot of the receiving angle vs. the instrument readings. The curves reach their maximum slope of 2.4 gloss units per degree at

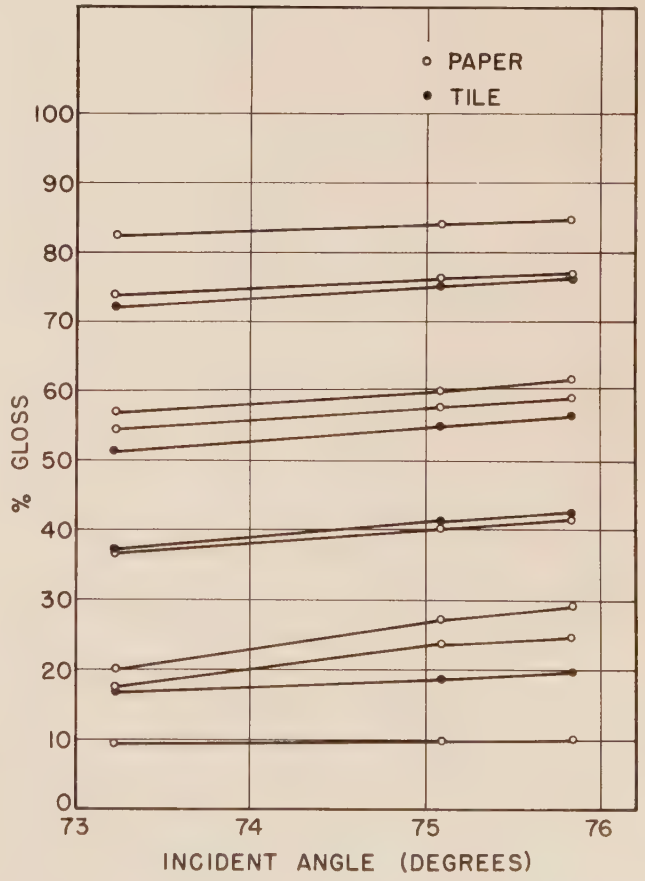



*Fig. 3. The effects of varying the incident angle*

**Table I. Description of Samples**

| Sample no. | Description |
|---|---|
| 1 | 25-lb. lightly coated book |
| 2 | 40-lb. supercalendered book |
| 3 | 40-lb. supercalendered book |
| 5 | 80-lb. machine coated book |
| 6 | 80-lb. machine coated enamel |
| 7 | 70-lb. machine coated enamel |
| 8 | Cast coated enamel |
| 9 | Cast coated enamel |
| 11 | Bureau of Standards tile no. H20D |
| 12 | Bureau of Standards tile no. H35C |
| 13 | Bureau of Standards tile no. H50C |
| 14 | Bureau of Standards tile no. H70C |

lower values of instrument reading than was the case when the incident angle was varied. An examination of the goniophotometric curves presented in Figs. 5 and 6 reveals that the maximum slope should occur at a lower gloss value. These curves are skewed toward angles larger than the incident angle, and moveover the angles of maximum reflection for the lower gloss samples are larger than the incident angle. Here, as in the case of incident angle variation, data obtained on the tiles were found to be more sensitive to receiving angle changes than were those on the papers. The flatter goniophotometric curves for the tiles would indicate this.

***The Receiving Angle Perpendicular to the Plane of Measurement***

For this experiment, the incident angle and receiving angle in the plane of measurement were set to be alike

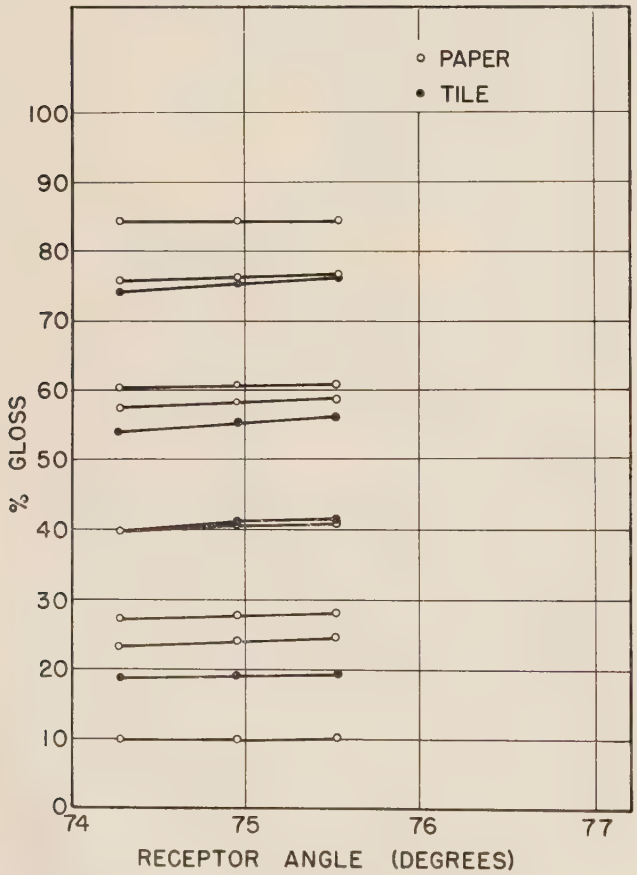



*Fig. 4. The effects of varying the receptor angle in the plane of the measurement*

and the receptor was moved perpendicular to the plane of measurement in three steps. The means of the four replications per sample are plotted against the deviation angle in Fig. 7. The slopes of these curves are not large; however, an analysis of variance shows them to be significantly different from zero to the 0.001 level. It is

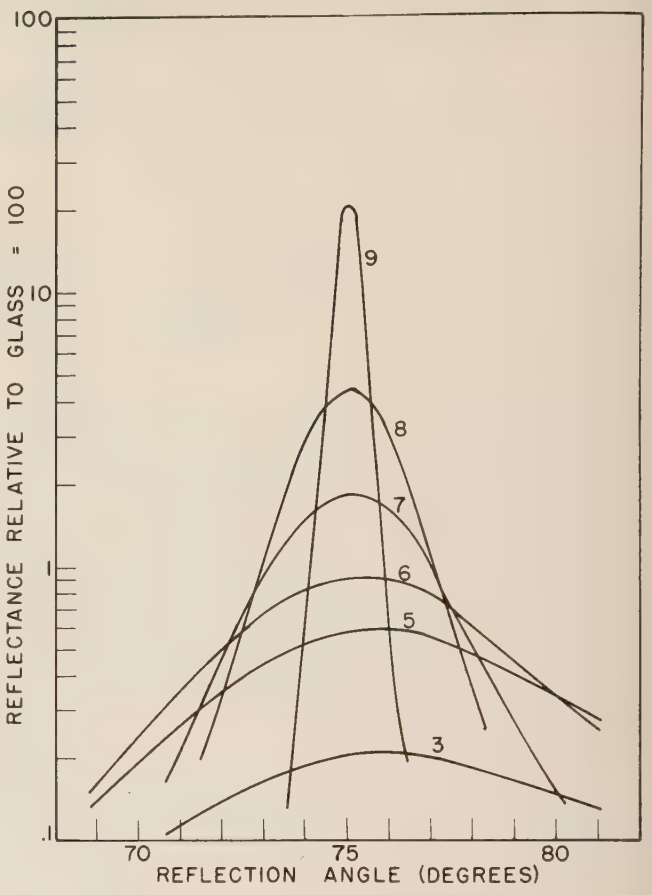



*Fig. 5. The effects of the angle of reflection on the intensity of the reflected light; the curves are numbered corresponding to the sample numbers*

estimated that the maximum slope for the tiles is of the order of −0.8 gloss unit per degree off center, which, it will be noted, is larger than that observed for the papers.

***The Diameter of the Receptor Aperture***

The diameter of the receptor aperture was varied in four steps. The resultant data are shown graphically in Fig. 8. The maximum slope of these curves is 1.65 gloss units per millimeter of aperture diameter. These curves show marked differences between the tiles and papers in their reaction to changes in aperture diameter. The differences are consistent with the goniophotometric data since the diameter of the receptor has a smaller effect on the gloss values of specimens having more slender goniophotometric curves. In the limit, the readings on a material such as the polished black glass standard are unchanged by changes in the receptor aperture diameter until the ratio of aperture diameter to image size reaches unity. Conversely, a perfect diffuse reflector would yield gloss values which are nearly a linear function of the receptor area.

***The Position of the Image of the Source***

To explore the effects of this variable, the image of the source was focused at two different positions, namely, at the center of the aperture and at 125 cm. beyond the aperture. The means of four measurements per sample are plotted against the position of the image in Fig. 9.

It is recognized that changing the position of the focus changes the convergence of the light and also changes the size of the light spot at the receptor. The total effect of these variables plus the variation caused by

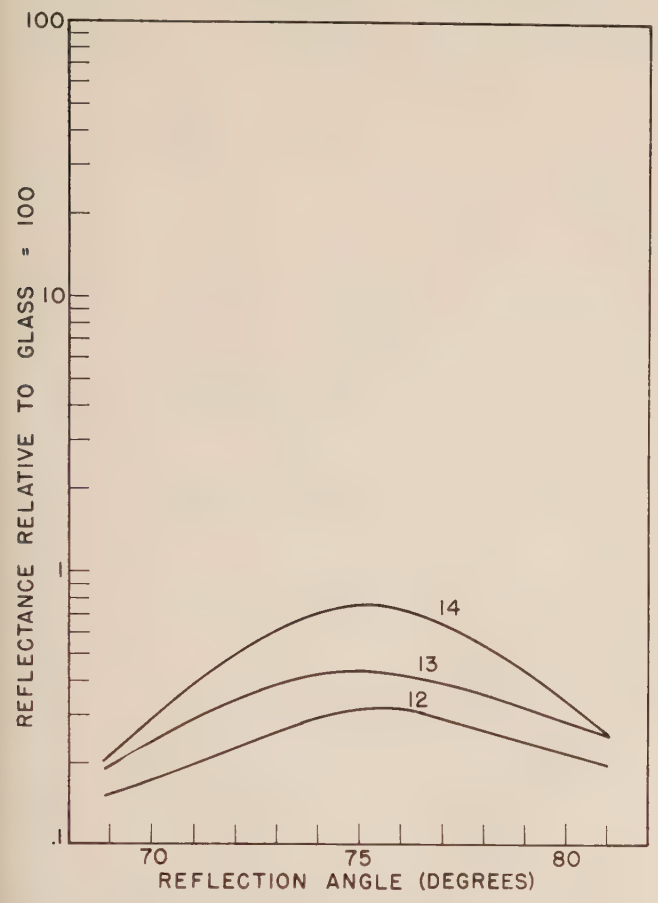



*Fig. 6. The effects of the angle of reflection on the intensity of the reflected light; the curves are numbered corresponding to the sample numbers*

variations in image position is only −0.015 unit of gloss per centimeter beyond the receptor.

### *The Image Size at the Receptor*

The size of the image of the source was varied by changing the lamp type at the source. Because of the simple optical system used, it was impractical to vary each dimension of the source independently. Thus, for these measurements, image width is confounded with image height and with beam convergence. The results are presented in Fig. 10 where it will be observed that the instrumental readings are plotted against the image width with the full knowledge of the inadequacy of such a presentation. If one assumes that all of the variation is caused by variations in image width, then the maximum slope of the curves is 0.5 gloss unit per millimeter of image width. In view of the relatively strong dependence of the instrumental readings on the size of the image, information was also obtained on the variability of filament size in lamps of the same type. For a series of ten G.E. lamps no. 1709, it was found that filament lengths varied from 1.66 to 1.95 mm. and diameters varied from 0.66 to 0.70 mm. The variations in size of the filament can give rise to variations in instrumental readings of as much as plus or minus 0.36 unit of gloss. Thus it is evident that an illuminated aperture would be preferred to the use of a direct filament as a source.

### *The Distance from the Test Area to the Lens*

To determine the effects of varying the distance from the lens to the center of the test area, lenses of different focal length were inserted in the lens tube. In order to maintain essentially the same image size, lamps with appropriate filament sizes were used to compensate for differences in lens magnification. Unfortunately such a procedure did not strictly maintain the image height constant, although the image width was maintained at a nearly constant value. The two lenses used resulted in lens to test area distances of 117 and 107 mm. An analysis of variance of the data indicated that the differences between the two conditions, if any, were not significant. There is the possibility that the effects of the increase in image width just compensated the effects of the decrease in image height. If this were the case, the foregoing conclusion would be valid. However, there is the possibility that image height has no effect on the readings and that the effects of the change in image width just offset a real variation caused by a change in the distance from the lens to the test area. In this case, the foregoing conclusion would be invalid. In view of the lack of a definitive experiment covering this variable, it would be desirable that this distance be

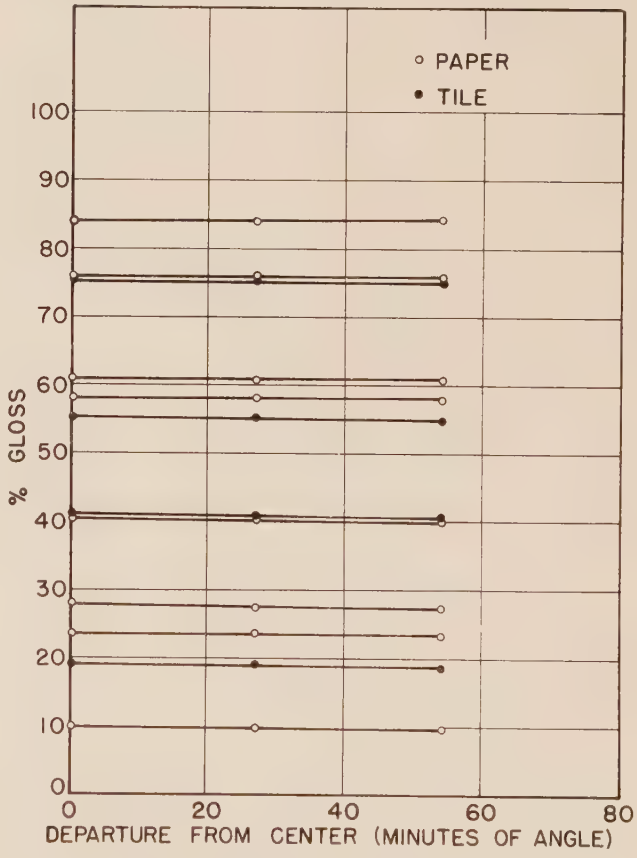



*Fig. 7. The effects of varying the receptor angle perpendicular to the plane of measurement*

held to some easily maintained tolerance well within the range of distances covered by the experiment.

### *The Source Aperture Size*

For this phase of the study, two sets of instrumental readings were obtained. In one the aperture width was maintained constant and the height varied, and in the

other the aperture height was maintained constant and the width varied. The three dimensions used were 15.8 by 10, 15.8 by 5.9, and 9.4 by 10 mm. The width, 15.8 and 9.4 mm., was measured perpendicular to the plane of the gloss measurement. When the data for 15.8 by 5.9 mm. were tested against that for 15.8 by 10 mm., using the analysis of variance technique, the analysis showed no significant difference. The conclusion to be drawn is either that the experiment was not sensitive enough to detect the dependence of instrument reading on aperture height or that there is no such dependence. In view of the very small dependence of instrumental reading on aperture height, no difficulty should be experienced in meeting this requirement in the construction of an instrument.

A similar analysis of the data for 15.8 by 10 and 9.4 by 10 mm. showed a small but significant dependence of the measured gloss on the width of the source aperture. The maximum slope for any single sample is −0.75 gloss unit per millimeter of aperture width.

***The Spectral Response***

The effects of varying the spectral response of the receptor were measured by inserting various filters in front of the receptor aperture. Three filters were used, namely (1) Viscor, (2) Corning Glass no. 556 melt 214,

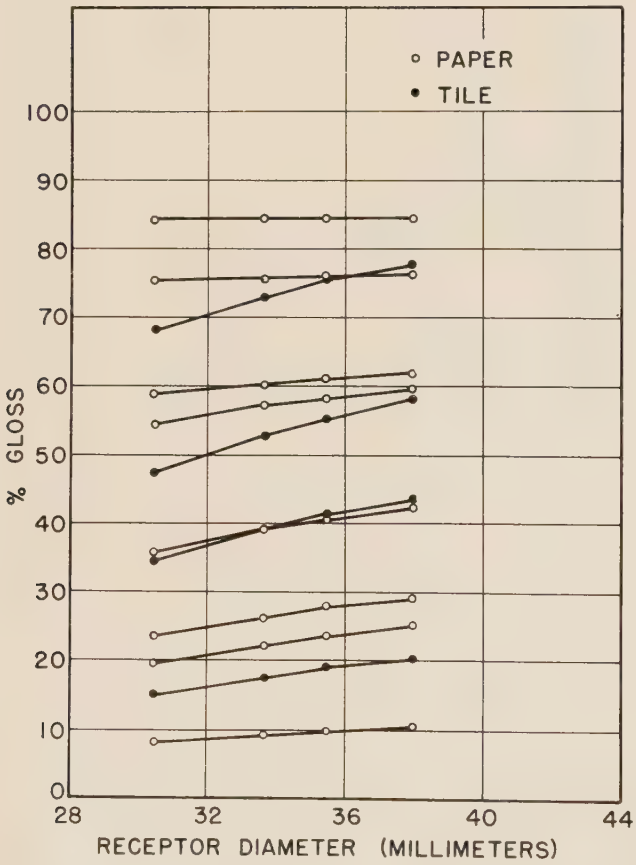



*Fig. 8. The effects of varying the receptor diameter*

and (3) Wratten E no. 22 in B glass. The normalized spectral response characteristics of the photocell in combination with each of the filters are shown in Fig. 11.* In use, the Corning blue filter and Wratten red filter were placed outside of the sphere in contact with the receptor aperture, whereas the Viscor filter was placed adjacent the photocell. Figure 12 is a plot of the instrumental readings as a function of wavelength. The curves show a very definite increase in readings with an increase in wavelength. The maximum slope reaches a

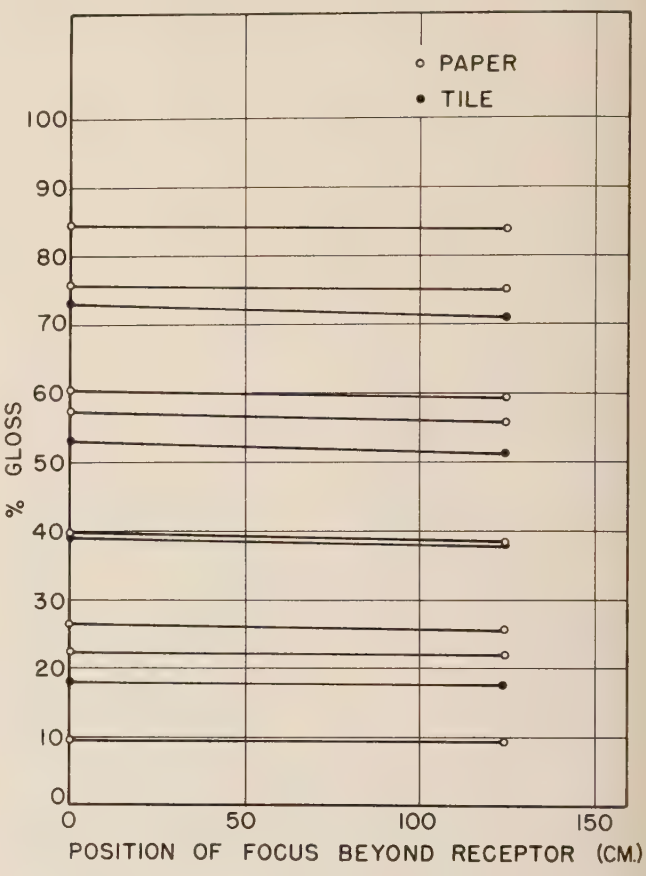



*Fig. 9. The effects of varying the position of focus of the image of the source*

value of 0.043 gloss unit per millimicron. This is the only case in the study in which the measured gloss of the paper samples is more sensitive to a given variable than that of the tiles. To attempt to obtain an explanation for the large dependence of indicated gloss on the wavelength of the light, goniophotometric curves were obtained on each of the samples, using 75° as the angle of incidence. Two sets of measurements were made with the goniophotometer using a filter and phototube combination that was close to but not identical to the green conditions (Fig. 11, curve B) previously used and the other approximating the blue curve (Fig. 11, curve A). Data obtained with the green filter are presented in Figs. 5 and 6. The data for the blue filter are not included because they were substantially identical to those obtained with the green filter. The differences between the two sets of goniophotometric data are not large enough to explain the differences obtained with the glossmeter when the spectral response was changed. There may be an off axis color effect that would account for the observed differences, but no attempt has been made to continue the investigation in this direction.

In a continuation of the study on spectral conditions, the color temperature of the source was varied by changing the lamp voltage. Two nominal voltages were used: four volts and six volts. Figure 13 shows the re-

* The response curve for the photocell and the transmission data for the Viscor filter were obtained from literature prepared by the Weston Electrical Instrument Co.

sults of changing lamp voltage. The lines connecting the two points for each sample are drawn as straight lines although the true curves are probably not straight. The differences in gloss resulting from a change in voltage are small, but an analysis of variance of the data indicates that these differences are significant. The

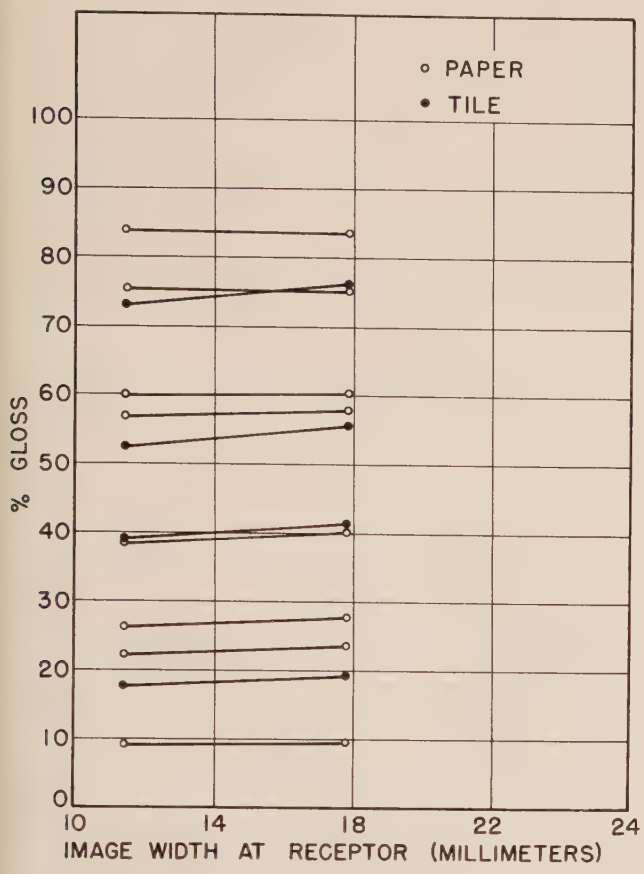



*Fig. 10. The effects of varying the image size*

maximum slope for any single sample is 0.42 gloss unit per volt change.

## SUMMARY AND CONCLUSIONS

The following is a summary showing the influence of the instrumental variables on gloss. The values which show the dependence of gloss on the various instrumental factors are, in each case, the maxima observed and are for an instrument in which the distance from the center of the sample opening to the receptor aperture is 166 mm.

**Table II**

| *Instrumental variable* | *Maximum variation* |
|---|---|
| Angle of incidence | 2.5 gloss units per degree |
| Receiving angle in the plane of measurement | 2.4 gloss units per degree |
| Receiving angle perpendicular to the plane of measurement | −0.8 gloss unit per degree |
| Diameter of the receptor aperture | 1.65 gloss units per millimeter |
| Position of the image of the source | −0.016 gloss unit per centimeter beyond the receptor |
| Image size | 0.5 gloss unit per millimeter of image width |
| Source aperture size | Noncritical in the plane of measurement<br>−0.075 gloss unit per millimeter in the plane perpendicular to that of measurement |
| Distance from the test area to the incident lens | Noncritical over a distance of 10 mm. |
| Spectral response | The product of the CIE luminosity and illuminant A distribution functions should be matched closely |

The gloss readings obtained for the tiles furnished by the National Bureau of Standards are, in general, more critically dependent on instrumental variables than are those for the papers. This is a desirable characteristic for gloss standards.

If it were assumed that all of the variables could contribute errors that add in the same direction and if it is desired to maintain the readings on several glossmeters within ±1% of full scale, then the results of this study indicate that the required instrumental tolerances should be those presented in Table III. The dimensions are in arbitrary units in which the distance from the center of the specimen to the center of the receptor aperture is taken as unity. Any given dimension of an instrumental variable may be obtained by multiplying the ratios listed in the table by the actual distance from the center of the specimen to the center of the receptor aperture.

The geometrical tolerances presented in Table III were derived on the basis of a practical glossmeter hav-

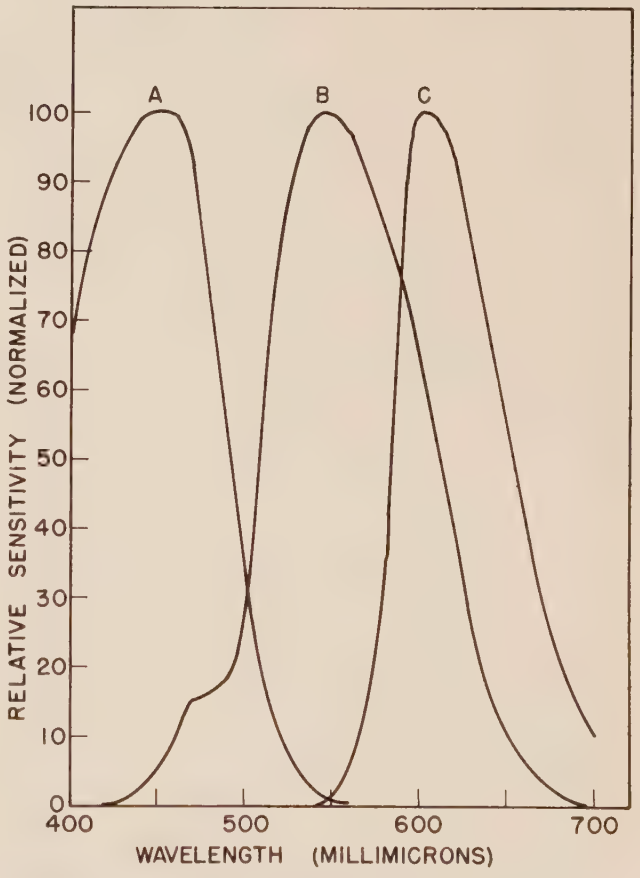



*Fig. 11. The spectral response of the filters plus the photronic cell*

A—Corning No. 556. B—Viscor. C—Wratten No. 22.

ing a specimen to receptor distance of approximately 160 mm. First, one half of the scale tolerance of ±1% of full scale was assigned to the meter and photocell. To reduce the linearity of the photocell-meter combination below ±0.5% is both expensive and difficult. If the diameter of the receptor aperture, the source aperture

size, the distance from the test area to the lens, the distance from the test area to the receptor and the size of the source ( a machine aperture) are all held to ±0.025 mm.—normal machine shop practice—then the total

**Table III**

| *Instrumental variable* | *Tolerances in TAPPI arbitrary units, dimensions, and ratios* |
|---|---|
| Angle of incidence | 75° ± 0.1° |
| Receiving angle in the plane of measurement | 75° ± 0.04° |
| Receiving angle perpendicular to the plane of measurement | 0° ± 0.08° |
| Diameter of receptor aperture | 0.2 ± 0.00012 |
| Position of the image of the source | At receptor aperture ±0.06 |
| Image size | 0.10 ± 0.0007 × 0.05 ± 0.0007 |
| Source aperture size | 0.25 ± 0.0006 × 0.15 ± 0.0006 |
| Spectral response | The product of the CIE luminosity and illuminant A distribution functions should be matched closely |
| Photocell and indicating meter | ±0 5% of full scale |

variation in reading arising from these sources will not be over ±0.1%. The receiving angle is determined by the position of the center of the receptor aperture with respect to the center of the specimen. Normal ma-

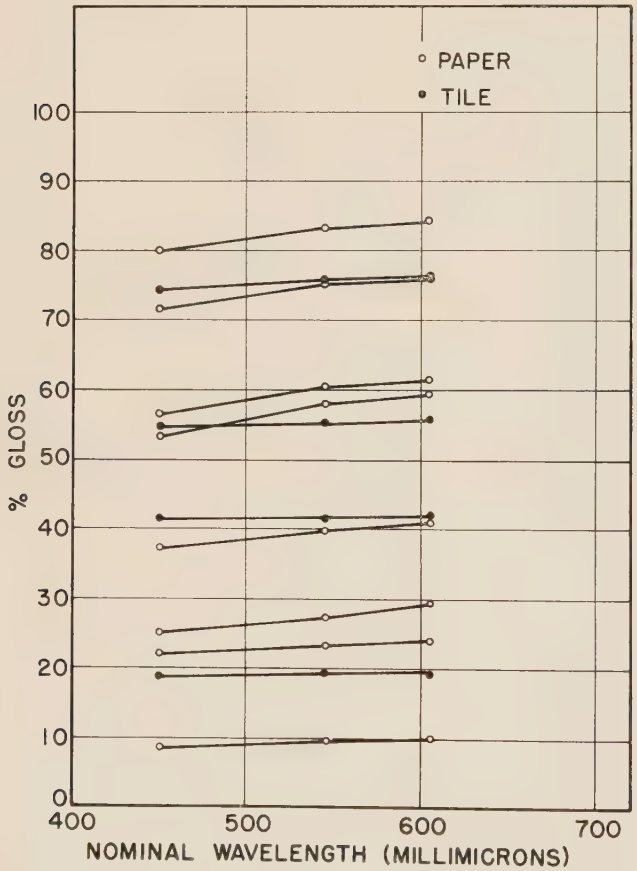



*Fig. 12. The effects of varying the spectral response*

chine shop practice will also permit this position to be held to ±0.025 mm., resulting in a variation of ±0.05% in the gloss reading. This leaves a residual of ±0.35% which may be divided between the angle of incidence and the spectral response. The incident angle tolerance of 0.1° is difficult but not impossible to hold. This tolerance cannot be made larger and permit any reasonable variation in the spectral characteristics.

The CIE luminosity function can be closely matched either with a Weston Photronic cell and a Viscor filter or with a photoemissive tube having an S-4 response and a Wratten no. 106 filter. The variations in reading

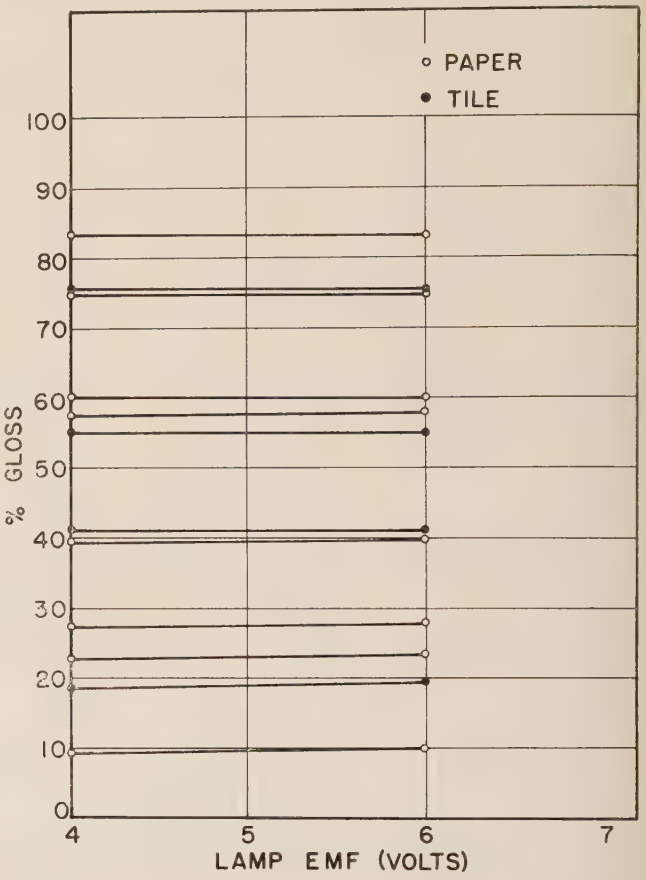



*Fig. 13. The effects of varying the source lamp voltage*

that can be ascribed to differences between photosensitive units and filters are unknown.

An instrument meeting the specifications listed in Table III has been constructed by Gaertner Scientific Corp. of Chicago, Ill. Preliminary tests on the new instrument indicate that it will function very satisfactorily.

## LITERATURE CITED


1. Hammond, H. K., and Nimeroff, I., *J. Res. N. B. S.* 44: 585 (1950), RP2105.
2. The Institute of Paper Chemistry Report No. 48 to The American Paper and Pulp Association. Study on Gloss, Part I, A Goniophotometric Study of High-Gloss Papers.



Received Dec. 21, 1953. Presented at the 39th Annual Meeting of the Technical Association of the Pulp and Paper Industry, New York, N. Y., Feb. 15–18, 1954.


## *Erratum—"The Effect of Different Pulps on the Biological Activity of Phenylmercuric Acetate," by B. F. Shema and J. H. Conkey*

The above paper was published in the July, 1954, issue of *Tappi*, page 294. On page 295 where it states "In another study (*10*), the same authors found that ethoxy ethyl mercury phosphate is adsorbed..." it should read "In another study ...that ethoxy ethyl mercury acetate is adsorbed...."

# ASSOCIATION NEWS AND EVENTS

## Local Section Meetings

*Pacific:* Nov. 9, 1954, Bellingham, Wash., "Instrumentation." Jan. 11, 1955, Longview, Wash., "What's New in Pulping."

*Kalamazoo Valley:* Nov. 4, 1954, "What Holds Paper Together," by H. O. Ware, Hercules Powder Co., Wilmington, Del.

*Southeastern:* Nov. 12–13, 1954, King & Prince Hotel, St. Simons Island, Ga. Speaker, Harry F. Lewis, Institute of Paper Chemistry, Appleton, Wis.

*Chicago:* Oct. 18, 1954, and Nov. 15, 1954, Chicago Bar Association, Chicago, Ill.

*Lake States:* Nov. 9, 1954, Riverside Country Club, Marinette, Wis. Visits to Badger Paper Mills and the Marinette Paper Co. mill. Demonstrations sponsored by the Ansul Chemical Co.

*New England:* Dec. 10, 1954, Roger Smith Hotel, Holyoke, Mass.

*Ohio:* Dec. 9, 1954, American Legion Hall, Middletown, Ohio. Speaker, G. H. Pringle, The Mead Corp., Chillicothe, Ohio.

*Empire State—Metropolitan District:* Nov. 9, 1954, Fraunces Tavern, New York, N. Y., "Quality Control in the Manufacture of Paperboard," by Joseph Theriault, Robert Gair Co., Uncasville, Conn.

*Empire State—Eastern District:* Oct. 21, 1954, and Nov. 18, 1954, Glens Falls, N. Y.

*Lake Erie:* Nov. 12, 1954, "The Application of Plastics in the Paper Industry." Dec. 11, 1954, Christmas Party.

*Empire State—Western District:* Nov. 3, 1954, joint meeting with Niagara Branch, Technical Section CPPA. Dec. 1, 1954, Prospect House, Niagara Falls, N. Y. Speaker, R. G. Macdonald, Secretary, TAPPI, New York, N. Y.

## Kalamazoo Valley

The Kalamazoo Valley Section has announced its schedule of meetings for 1954–55. All meetings will be held in the Hotel Harris, Kalamazoo, Mich.

*Sept. 30, 1954:* "Theoretical Approach to the Problems of Multidrying Cylinders," by Alfred H. Nisson, The University, Leeds, England.

*Nov. 4, 1954:* "What Holds Paper Together," by H. O. Ware, Hercules Powder Co., Wilmington, Del.; "Hydration and Bonding," by Robert T. Elias, Western Michigan College, Kalamazoo, Mich.; and "Bonding by Additives," by Frank Kaulakis, Clinton Foods, Inc., Clinton, Iowa.

*Dec. 2, 1954:* "New Testing Methods," discussion led by Paul D. Applegate, Hercules Powder Co., Kalamazoo, Mich.

*Jan. 13, 1955:* Joint meeting with the Michigan Div., APPM Superintendents Association.

*Feb. 3, 1955:* "The Technical Man Looks at Practical Mill Problems"—"Bleaching," by Stephen Kukolich, Lee Paper Co., Vicksburg, Mich., and "Stock Treatment," by W. F. Hathaway, Kalamazoo Vegetable Parchment Co., Kalamazoo, Mich.

*March 3, 1955:* Graphic Arts Meeting, Peter Moffett, Kalamazoo Vegetable Fibre Co., Kalamazoo, Mich., chairman.

*April 7, 1955:* Engineering Meeting, D. B. Gearhart, General Electric Co., Kalamazoo, Mich., chairman.

*May, 5, 1955:* Western Michigan College, Senior Theses Presentations; "What Lies Ahead?," by Elmer K. Stilbert, Jr., Dow Chemical Co., Midland, Mich.

*June 1955:* Annual meeting, Fun Day at Gull Lake.

## Delaware Valley

The Delaware Valley Section has announced its schedule of meeting dates for 1954–55. Unless otherwise indicated all meetings are held at the Engineers Club, Spruce St., Philadelphia, Pa.

*Sept. 16, 1954:* Visit to mill of Scott Paper Co., Chester, Pa. Meeting at the Chester Club House. "The Theoretical Aspects of Drying Paper on Fourdrinier and Cylinder Machines," by Alfred Nisson, Professor of Textile Engineering, The University, Leeds, England.

*Oct. 18–21, 1954:* TAPPI Ninth Engineering Conference, Benjamin Franklin Hotel, Philadelphia, Pa.

*Dec. 10, 1954:* Panel Discussion—"Latest Developments in Beater Additives."

*Jan. 27, 1955:* Graphic Arts Meeting, joint meeting with the Printing Ink Makers Production Club (Panel discussion).

*March 24, 1955:* "Paper Mill Management," by K. O. Elderkin, Bowaters Southern Paper Corp., Calhoun, Tenn.

*April 28, 1955:* Panel discussion on "Stock Preparation."

*May 26, 1955:* Edward J. Albert Award Competition.

## Empire State—Northern District

The Northern District of the Empire State Section has announced its schedule. Meetings will be held in the Woodruff Hotel, Watertown, N. Y.

*Oct. 14, 1954:* General discussion of products of Northern New York Mills.

*Nov. 11, 1954, Dec. 9, 1954, Jan. 13, 1955, Feb. 10, 1955, March 10, 1955, April 14, 1955:* Empire State Award Papers.

*May 12, 1955:* Annual meeting.

## Pacific

The 1954–55 schedule of meetings for the Pacific Section has been established as follows:

*Sept. 29, 1954:* Multnomah Hotel, Portland, Ore., engineering program.

*Nov. 9, 1954:* Bellingham, Wash., instrumentation program.

*Jan. 11, 1955:* Longview, Wash., pulping program.

*March 4, 1955:* Everett, Wash., Shibley Award program.

*May 18–21, 1955:* Victoria, B. C., joint meeting with Pacific Branch, Technical Section, CPPA, and Pacific Div., APPM Superintendents Association

## Lake Erie

The Lake Erie Section has announced the following schedule of its meetings for 1954–55. All meetings will be held on the second Friday of each month at the Hotel Carter, Cleveland, Ohio.

*Sept. 10, 1954:* "Time and Motion Study and Incentive Measurement."

*Oct. 8, 1954:* "Board vs. Light Weight Kraft as It Affects Material Suppliers."

*Nov. 12, 1954:* "The Application of Plastics in the Paper Industry."

*Dec. 11, 1954:* Christmas Party.
*Jan. 14, 1955:* "Materials Handling."
*Feb. 11, 1955:* "Industrial Psychology."
*March 11, 1955:* "Measurement and Control in the Graphic Arts."
*April 15, 1955:* "Recent Developments in Printing Inks."
*May 13, 1955:* Open.

## Empire State—Metropolitan District

The schedule of meetings of the Metropolitan District has been announced as follows (all meetings will be held at Fraunces Tavern, New York, N. Y.):

*Sept. 14, 1954:* "Practical Examples in the Development of Paper Coatings," by Walter L. Hardy, Foster D. Snell, Inc., New York, N. Y.
*Oct. 19, 1954:* "Principles in Corrugated Shipping Container Design," by Theodore J. Gross, Union Bag & Paper Corp., New York, N. Y.
*Nov. 9, 1954:* "Quality Control in the Manufacture of Paper Board," by Joseph Theriault, Robert Gair Co., Uncasville, Conn.
*Dec. 14, 1954:* Panel discussion, "Customer Demands and Sales and Manufacturing Problems Arising Therefrom."
*Jan. 11, 1955:* "Comparison of Various Pulps from a Use Aspect" by Gerald Haywood, West Virginia Pulp & Paper Co., Luke, Md.
*March 8, 1955:* Panel discussion on "Printing."
*April 12, 1955:* "Fundamentals of Stock Preparation."
*May 10, 1955:* Annual meeting and Ladies Night.

## New England

The New England Section has announced its schedule of 1954–55 meetings as follows:

*Oct. 8–9, 1954:* Berkshire Inn, Great Barrington, Mass., visits to mills of Crane & Co. and Byron Weston Co., Dalton, Mass.
*Dec. 10, 1954:* Roger Smith Hotel, Holyoke, Mass.
*March 25, 1955:* Statler Hotel, Hartford, Conn.
*June 3–4, 1955:* Oceanside Hotel, Magnolia, Mass.

## Empire State—Eastern District

The meetings of the Eastern District will be held on the third Thursday of each month from October to April. The May, 1955, meeting will be held on Saturday, May 21.

## Container Testing Committee

Milton Yezek, manager of research and quality control, Carton and Container Div., General Foods Co., Battle Creek, Mich., has succeeded Charles H. Krebs, Atlas Boxmakers, Inc., Chicago, Ill., as chairman of the TAPPI Container Testing Committee.

***Milton Yezek, General Foods Co.; Chairman, Container Testing Committee***

## Pulp Testing Committee

The TAPPI Pulp Testing Committee is concerned primarily with the physical evaluation of all kinds of pulp. At present there are 14 methods under this committee's jurisdiction. Some of these methods such as the beater method for evaluating pulp, dirt in pulp, along with new procedures, are under study. Future plans include a comprehensive program to develop new pulp testing methods.

Members of the Technical Association who are interested in pulp testing are invited to apply to the Secretary of TAPPI, 155 E. 44th St., New York 17, N. Y., for appointment as members of the Pulp Testing Committee.

## Mechanical Pulping Conference

The TAPPI Mechanical Pulping Committee and the CPPA Technical Sections Groundwood Committee will sponsor jointly a four-day International Mechanical Pulping Conference to be held at the Poland Spring House, Poland Spring, Me., on Sept. 19–21, 1955.

A preliminary survey of subjects to be considered at the conference indicates an interest in "Regional Trends in Groundwood Manufacture" and "Guiding Techniques." Many other topics were recommended for consideration.

A meeting to work out plans for the meeting was held recently in Montreal, P. Q., and was attended by W. H. de Montmorency, Pulp & Paper Research Institute of Canada, Montreal, P. Q., T. F. LaHaise, Imperial Paper & Color Corp., Plattsburg, N. Y., J. H. Perry, Norton Co., Worcester, Mass., R. A. Joss, Technical Section, CPPA, Montreal, P. Q., and A. J. Winchester, TAPPI, New York, N. Y.

F. W. O'Neil, College of Forestry, State University of New York, Syracuse, N. Y., is chairman of the TAPPI Mechanical Pulping Committee. J. H. Perry, Norton Co., is secretary. The members of the committee include:

Axel Hyttinen, Forest Products Laboratory, Madison, Wis.
E. H. Johnson, Stevens & Thompson Paper Co., Greenwich, N. Y.
J. K. Kirkpatrick, Bowaters Southern Paper Corp., Calhoun, Tenn.
T. F. LaHaise, Imperial Paper & Color Co., Plattsburg, N. Y.
J. H. Heuer, Great Northern Paper Co., Millinocket, Me.
T. E. Kloss, Hollingsworth & Whitney Co., Waterville, Me.
R. S. Magruder, Sprout, Waldron Co., Muncy, Pa.
W. H. de Montmorency, Pulp & Paper Research Institute of Canada, Montreal, P. Q.
N. F. Robertson, West Tacoma Newsprint Co., West Tacoma, Wash.
J. H. White, Keyes Fibre Co., Waterville, Me.
K. L. Pingrey, Diamond Match Co., Ogdensburg, N. Y.

## Research Appropriations Committee

John L. Parsons, chairman of the TAPPI Research Appropriations Committee, reports the following requests for Association funds to finance research projects between January and July, 1954:

No. 66. "Vapor Equilibrium Data for Systems Magnesium Oxide–Sulphur Dioxide–Water and Sodium Oxide–Sulphur Dioxide–Water. Requested by the TAPPI Acid Pulping Committee for work to be carried on at the University of Washington, Seattle, Wash. ($2500).

No. 67. "Statistical Analysis of Digester Wall Thickness Measurements." Requested by the Digester Corrosion Subcommittee of the TAPPI Chemical Engineering Committee for work to be done at the Armour Research Institute, Illinois Institute of Technology, Chicago, Ill. ($3000).

No. 68. Study on "Isolation of Dimers from Lignin." Requested by TAPPI Fundamental Research Committee ($4500).

No. 69. "Printer-Slotter Creaser Contour Study." Requested by the TAPPI Corrugated Containers Committee for work to be conducted at The Institute of Paper Chemistry, Appleton, Wis. ($6500).

The total amount of requests granted is $12,000.

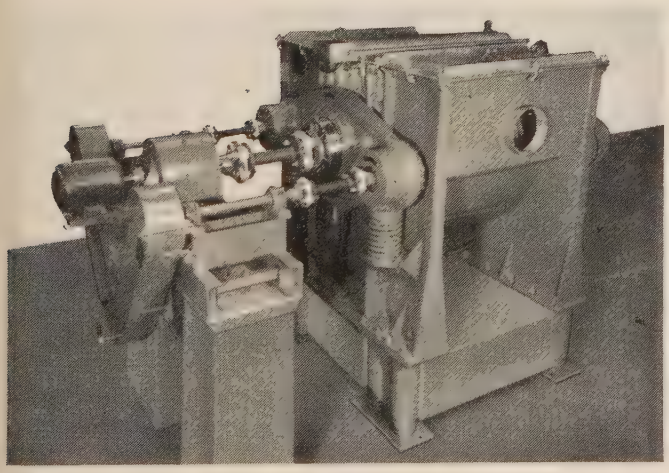
Request No. 68 was not approved unanimously by the Research Appropriations Committee but did receive majority approval. This request has been referred to the TAPPI Executive Committee for final disposition.

During the period formerly to June, 1953, applications for

| Type of Stock | Screen Plate | Consistency | Tonnage Per Day |
|---|---|---|---|
| Sulphite, unbleached | .010″ | 1.5% | 45 |
| | .012″ | 1.5 | 52 |
| bleached | .010″ | 1.7 | 54 |
| | .012″ | 1.5 | 65 |
| | .014″ | 1.6 | 75 |
| Sulphate, unbleached | .012″ | 1.5-1.9 | 55-68 |
| Gum, unbleached | .012″ | 1.5 | 110 |
| Board, semi-chemical | .018″ | 1.7 | 86 |
| | .035″ | 1.6 | 150 |
| De-Ink Paper Stock | .010″ | 2.2 | 110 |
| | 012″ | 1.9 | 148 |
| News Print | .014″ | 1.7 | 83 |

*Look at this record*

# OF BIRD VIBROTOR SCREEN PERFORMANCE



These are average consistencies handled and tonnages produced by Vibrotor Screens with 40-inch screen cylinders. Higher consistencies are often screened and larger tonnages often produced.

Whatever your screening problem, it will pay you to find out what the Vibrotor Screen can do and how much it may save in space and in screening cost per ton.

## BIRD MACHINE COMPANY

SOUTH WALPOLE • MASSACHUSETTS

Association funds amounted to $27,100 covering 12 committee requests.

One request received in July, 1954, is now being considered by the Research Appropriations Committee. This request relates to the project on disperse cellulose viscosity, sponsored by the TAPPI Chemical Methods Committee.

A survey of research and testing facilities in more than 30 organizations throughout the country, for carrying out TAPPI committee research projects, has been made by the Research Appropriations Committee. These results were distributed to every chairman in the TAPPI committee organization on June 7, 1954.

## *TAPPI Annual Meeting 1955*

The *Fortieth Annual Meeting* of the Technical Association of the Pulp and Paper Industry will be held at the Commodore Hotel, New York, N. Y. on Feb. 21–24, 1955. Individuals or companies desiring to present papers should advise the Secretary of the Technical Association immediately. Manuscripts are due on Dec. 1, 1954.

The following preliminary program indicates the sessions that have been reserved to date:

*General Session*

1. "Architectural Trends for Pulp and Paper Mills," by Mario C. Celli, Celli-Flynn, Inc., McKeesport, Pa.
2. "Facilities for Pulp and Paper Research at Lowell Technological Institute," by John Lewis, Dept. of Paper Engineering, Lowell Technological Institute, Lowell, Mass.

*Microbiological*

1. "Recent Developments in the Use of Chemical Treatments and Synthetic Fibers in Paper Machine Felts."

*Structural Fibrous Materials*

1—Three-hour session.

*Deinking*

1. "The Ahlfors Screen." by F. Schorken, Oliver United Filters, Inc., Oakland, Calif.
2. "Quality Problems in Waste Paper Consumption."

*Cylinder Board*

2—Three-hour sessions.

1. "Vat Exhauster Survey," by Otis Whitworth, Black-Clawson Co., Hamilton, Ohio.
2. "Development of the Shartle Alligator Refiner," by H. P. Espenmiller, Black-Clawson Co., Middletown, Ohio.

*Water*

1—Three-hour session.

*Fundamental Research*

1—Three-hour session.

*Acid Pulping*

1 —Three-hour session.

*Chemical Engineering*

1—Three-hour session.

*Dissolving Pulps*

2—Three-hour sessions.

1. "Resistance of Cellulose and Hemicelluloses to Hydrolysis," by R. L. Mitchell and J. F. Saeman, Forest Products Laboratory, Madison, Wis.
2. "A Method for Qualifying Dissolving Grade Pulps," by B. G. Hoos, Brown Co., Berlin, N. H.
3. "Viscose Rayon Tire Cord," by W. E. Roseveare, E. I. du Pont de Nemours, Inc., Richmond, Va.
4. Dissolving Pulp Symposium.

*Statistics*

2—Three-hour sessions.

*Plastics*

1—Three-hour session.

*Graphic Arts*

1—Three-hour session.

*Patents*

1. "Patent Office Practice," by Frank Makara, New York, N. Y.
2. "Recent Court Decisions," by Frank Makara, New York, N. Y.

*Engineering*

1. "Graphical Determination of Roll Deflections," by R. C. Sturken, Frank W. Egan Co., Bound Brook, N. J.
2. "Pneumatic Clutches in the Paper Mill," by J. S. Walsh, Federal Fawick Corp., Cleveland, Ohio.

*Fourdrinier*

1. "A New Concept in Roll Wrapping," by C. S. Adams, Rice Barton Corp., Worcester, Mass.
2. "Electrokinetics of Rosin Size," University of Maine paper.
3. "Dimensional Stabilization," by H. Ericks, The Upson Co., Lockport, N. Y.
4. "The Deculator-Cleaner," by J. C. Stewart and J. A. Smith, The Rotareaed Corp., Bronxville, N. Y.
5. "The Sulzer System of Water Extraction for Paper Machines," by Robert Thomann, Sulzer Bros., Winterthur, Switzerland.

## *TAPPI Fibrous Agricultural Residues Conference*

**The TAPPI Fibrous Agricultural Residues Conference dealing with straw pulping and papermaking, scheduled to be held on Nov. 8–9, 1955 in Peoria, Ill., will not be held.**

**S. I. Aronovsky, Chairman of the Conference, has found it necessary to enter the Mayo Clinic and does not expect to be available to conduct the conference as he has done in the past.**

**The next meeting of the committee will be held at the Commodore Hotel in New York City on Feb. 21, 1955.**

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**Corrugated Containers Conference**
**of the**
**Technical Association**
**of the**
**Pulp and Paper Industry**
**General Oglethorpe Hotel**
**Savannah, Ga.**
**Nov. 17–19, 1954**

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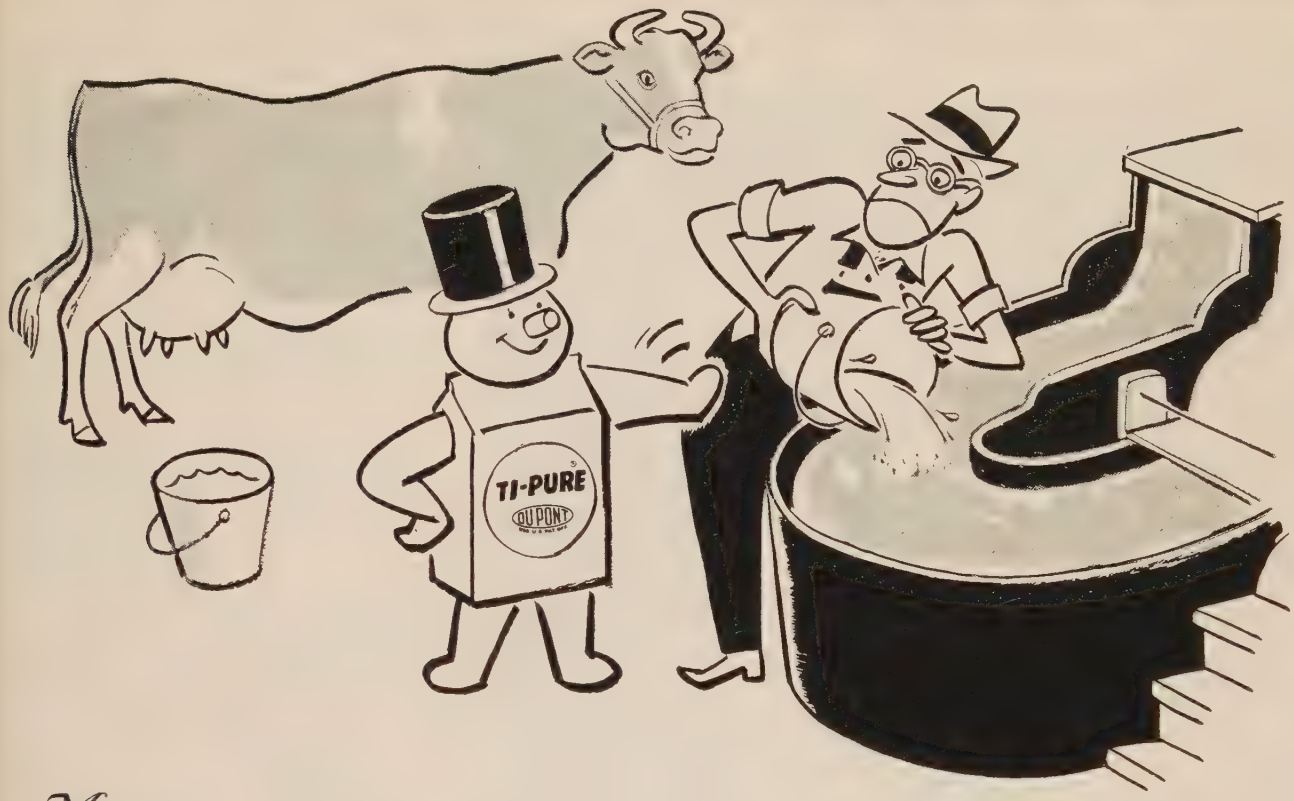
# *"Milk in your paper just won't do, Let **Ti-Pure**® whiten it for you!"*




*Mill owner Samson was all in a stew,*
*Dark, dingy paper was making him blue.*
*His pulp was, at best, a sad dreary lot,*
*Till TI-PURE of DU PONT arrived on the spot—*

*"Throw out that milk, Sam, don't be a back-seater!*
*It's good-by to dark paper, with me in your beater.*
*Like all Du Pont pigments, I've been put to the test,*
*As a filler for paper, I'm considered the best!"*

*Now Sam uses TI-PURE—he's oft heard to say,*
*"My paper's the whitest in many a day."*

***MORAL:***

*Why you should use TI-PURE is quite plain to see:*
*It gives whiteness . . . and brightness . . . and opacity!*

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Du Pont now offers a direct-to-you field service on TI-PURE titanium dioxide pigments. Technical experts, backed by a modern paper laboratory, will be glad to help you with your pigmenting problems. Call our nearest office for details.

**PROMPT, NATION-WIDE SERVICE THROUGH THESE DU PONT DISTRICT OFFICES* AND WAREHOUSES:**

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Minneapolis, Minn.
New Orleans, La.
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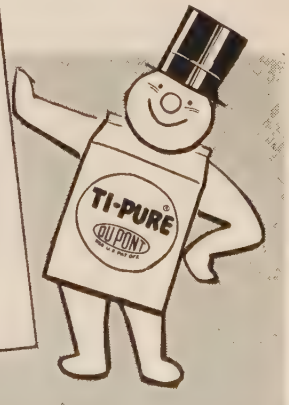
**Ti-Pure**® TITANIUM DIOXIDE

**TI-PURE gives your paper:**

**WHITENESS.** Lasting whiteness in food wraps and containers, waxed board, glassine, and parchment.

**BRIGHTNESS.** Permits sharp reproduction of printed matter in porous papers, such as magazine and book stock.

**OPACITY.** No "show-through" in lightweight papers, envelope and stationery stock.

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# PERSONAL MENTION

## *Items About New and Old Tappimen*

### *New TAPPI Members*

*David P. Antos*, Instrumentation Engineer, International Paper Co., Glens Falls, N. Y., a 1945 graduate of the New York State College of Forestry.

*George S. Armstrong*, President, George S. Armstrong & Co., Inc., New York, N. Y., a 1908 graduate of New York University.

*Rolland A. Aubey*, Development Supervisor, Nekoosa-Edwards Paper Co., Port Edwards, N. Y., a 1949 graduate of the University of Wisconsin.

*George W. Bremer*, Head of Research Dept., Cesterreichische Chemische Werke G.m.b.H., Weissenstein, Austria, a 1948 graduate of the University of Vienna with a Ph.D. degree.

*Ralph F. Carter*, Assistant Pulp Mill Superintendent, St. Marys Kraft Corp., St. Marys, Ga.

*Elizabeth L. Case*, Engineer, G. D. Jenssen Co., Inc., Watertown, N. Y., a 1951 graduate of Syracuse University.

*Andrew J. Chase*, Assistant Professor of Chemical Engineering, University of Maine, Orono, Me., a 1949 graduate of the University of Maine.

*Earl H. Donley*, Sales Engineer, The Babcock & Wilcox Co., New York, N. Y. Attended Pennsylvania State College.

*John W. Eastes*, Research Project Leader, Scott Paper Co., Chester, Pa., a 1936 graduate of the University of Cincinnati with a Ph.D. degree.

*John H. Ernlund*, Chemist, Minnesota Mining & Mfg. Co., St. Paul, Minn., a 1951 graduate of the University of Washington.

*Bernard F. Gentsch*, Vice-President and Sales Manager, United Clay Mines Corp., Trenton, N. J., a 1940 graduate of Alfred University.

*Goran Gyllenkrok*, Production Manager, Djupafors Fabriks AB, Kallinge Station, Sweden.

*Preben Hansen*, Paper Wholesaler, Thisted, Denmark.

*George H. Hellenberg*, Director of Laboratory, G. H. Puhrmann's Papiergroothandel N.V., Amsterdam, Netherlands, a 1951 graduate of the Technical University, Delft.

*Robert F. Hoban*, Assistant Head of Paper Dept., Sandoz Chemical Works, Inc., New York, a 1954 graduate of the New York State College of Forestry.

*Fred M. Howard*, General Manager, The Modene Paint Co., Inc., Chelsea, Mass., a graduate of the University of Amsterdam, The Netherlands.

*Leslie W. Hutchins*, Manager, Industrial Sales, Esso Standard Oil Co., Boston, Mass., a 1922 graduate of the University of Maine.

*John G. Kauffman*, Plant Engineer, Michigan Paper Co., Plainwell, Mich., a 1948 graduate of Drexel Institute of Technology.

*William R. James*, Chemist, Monsanto Chemical Co., St. Louis, Mo., a 1947 graduate of Pennsylvania State College with a Ph.D. degree.

*William F. Malcolm*, Sales Representative, Titanium Pigment Corp., Boston, Mass., a 1937 graduate of Alma College.

*Edmund McCarthy*, Consulting Engineer, Pocahontas Fuel Co., Salem, Mass., a 1927 graduate of Pratt Institute.

*Ralph E. Miller, Jr.*, Chemical Engineer, Union Bag & Paper Corp., Savannah Ga., a 1944 graduate of the University of South Carolina.

*Dipiendra N. Mitra*, Works and Development Chemist, Millspaugh Ltd., Sheffield, England, a 1949 graduate of the College of Engineering & Technology, Jadavpur, India.

*Eli F. Nicosia*, Chemist, Naval Air Station, Pensacola, Fla., a 1953 graduate of the University of Alabama.

*Dudley J. Nixon*, Technical Sales, Geigy Dyestuffs, Div. of Geigy Chemical Corp., New York, N. Y., a 1951 graduate of Pratt Institute School of Science & Technology.

*Robert J. Pavlin*, Sales Representative, E. I. du Pont de Nemours & Co., Inc., New York, N. Y., a 1947 graduate of the University of Notre Dame.

*John C. Pevear*, Paper Mill Mechanical Engineer, West Virginia Pulp & Paper Co., Luke, Md., a 1945 graduate of Tufts College.

*John G. Preston*, Adhesive Engineer, Federal Adhesives Corp., Brooklyn, N. Y.

*Earl F. Riter*, Manager, Soya Products Dept., The Glidden Co., Chicago, Ill., a 1930 graduate of Ohio University.

*Arthur R. Root, Jr.*, Research Chemical Engineer, Champion Paper & Fibre Co., Hamilton, Ohio, a 1954 graduate of the University of Cincinnati.

*Laurence E. Rossiter*, New England Sales Manager, Nopco Chemical Co., Boston, Mass., a 1934 graduate of New Bedford Institute of Textiles and Technology.

*Helen Rowley*, Librarian, E. I. du Pont de Nemours & Co., Inc., Textile Fibers Dept., Wilmington, Del., a 1931 graduate of Wells College.

*George H. Saaf-Norden*, Head, Technical Div., Austrian Pulp and Paper Association, and Austrian Paper and Paperboard Cartel, Vienna, Austria, a 1937 graduate of the Technical University, Vienna.

*Pier G. Sandri*, Maintenance Engineer, Cartiere Burgo S.p.A., Torino, Italy, a 1950 graduate of the Polytechnic University of Torino.

*George W. Sargent, Jr.*, Staff Engineer, The Ruberoid Co., South Bound Brook, N. J., a 1948 graduate of Massachusetts Institute of Technology.

*David G. Sheffield*, Manager, Cotton Linter Div., The Railway Supply & Mfg. Co., Memphis, Tenn.

*Robert E. Simkins*, Professional Engineer, Mattson, Putnam & Simkins, Seattle, Wash., a 1936 graduate of the University of Michigan.

*Benjamin F. Sproat*, Board Sales Service Representative, Mead Corp., Chillicothe, Ohio, a 1948 graduate of the University of Michigan.

*William H. Stine*, Research Engineer, The Champion Paper & Fibre Co., Hamilton, Ohio, a 1950 graduate of the University of Cincinnati.

*Thomas F. Sullivan*, Quality Control Supervisor, Hartford City Paper Co., Hartford City, Ind., a 1954 graduate of the New York State College of Forestry.

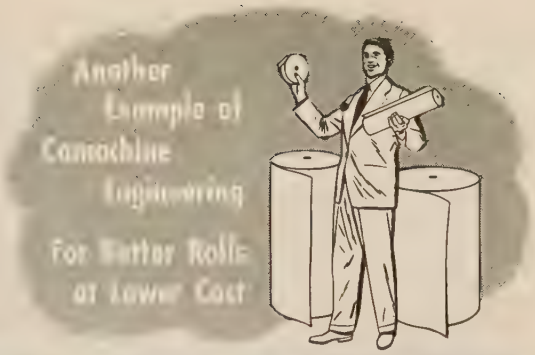
*Wilbur J. Sundheimer*, Manager, Engineering Dept., General Electric Co., Grand Rapids, Mich., a 1948 graduate of Purdue University.

*Yurugi Tadashi*, Pulp and Rayon Research, The Textile Research Institute of the Japanese Government, Yokohama, Japan, a 1940 graduate of the Applied Chemical Institute of the Tokyo Imperial University.

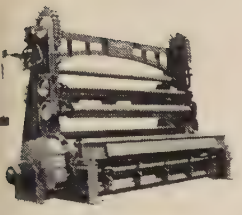
*Jennings P. Ward*, Tour Foreman, Pulp Mill, North Carolina Pulp Co., Plymouth, N. C.

*Chien Jo Yeh*, Graduate Student, Dept. of Chemical Engi-

# Hydraulic RIDING ROLL LIFT

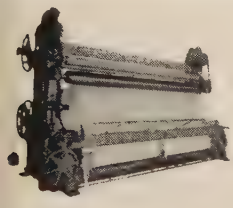



**Smooth-acting,** balanced control with no manual effort. Hydraulic cushioning prevents shock at the maximum upward-travel point of the piston and assures impact-free contact between riding roll and rewind shaft at the start of winding. Automatic operation reduces down-time between sets.



## CAMACHINE TYPE 20

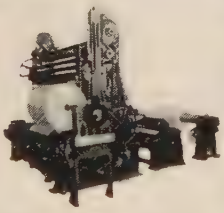
Widths to 310". Maximum rewind capacity may be either 42", 60" or 72" dia. Speeds up to 5,000 fpm* on paper and paperboard.



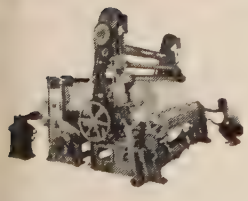
## CAMACHINE TYPE 19

Widths to 192". Maximum rewind capacity may be either 42", 60" or 72" dia. Speeds up to 3,500 fpm* on paper and paperboard.

## CAMACHINE TYPE 18



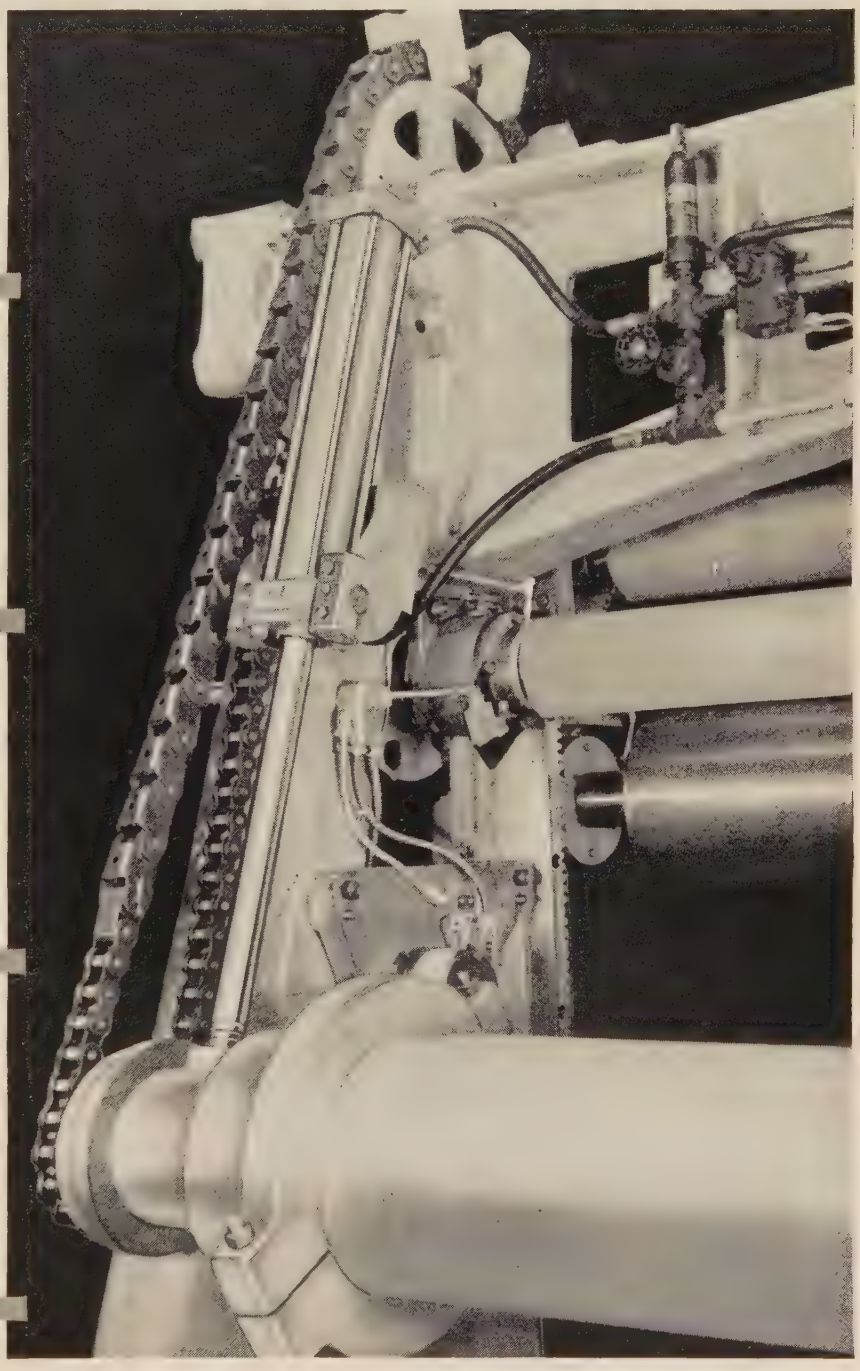
Widths to 150". Maximum rewind capacity may be either 42", 60" or 72" dia. Speeds up to 2,500 fpm* on paper and paperboard.



## CAMACHINE TYPE 14

Widths to 92". Maximum rewind capacity may be either 38", 50" or 60" dia. Speeds up to 1,800 fpm* on paper and paperboard.

*Speeds depend upon characteristics of material and machine width.



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AA-287

neering, University of Maine, Orono, Me., a 1952 graduate of the National Taiwan University.

*Hugo G. Zwecker*, Apprentice Papermaker, Papierfabrik Wattens, Wattens, Tyrol, Austria, a 1953 graduate of Technical College, Moedling.

## *TAPPI Notes*

*Russell L. Adams, Jr.*, formerly of the Riegel Carolina Corp., is now Chemical Engineer for the West Virginia Pulp & Paper Co., Luke, Md.

*Tore H. Ahlen*, formerly of A/B Svensk Flaktfabriken, is now associated with the Lundberg-Ahlen Equipment Co., Vancouver, B. C.

*John W. Bard* is now Manager of the Marathon Corp., Green Bay, Wis.

*Otto Berggren*, formerly of Personal Products Corp. is now with Stonebridge Paper Co., Wilington, Ill.

*Leslie K. Bickell* is now Technical Service Director for Alaska Pine & Cellulose Ltd., Vancouver, B. C.

*Oscar C. Brauner*, formerly of Hollinghead Corp., is now Chief Chemist for Sanders Chemical Co., Philadelphia Pa.

*John Q. Burton* is now Assistant Director of Research for the Minnesota & Ontario Paper Co., Minneapolis, Minn.

*Benton R. Cancel* is now President of the Rhinelander Paper Co., Rhinelander, Wis.

*Henry R. Chope*, is now Vice-President of Industrial Nucleonics Corp., Columbus, Ohio.

*Harry G. Davis*, formerly of the Oxford Paper Co., is now General Manager of Tube Turns Plastics, Inc., Louisville, Ky.

*Alvara de la Macorra* is now Technical Director of Papelera Nacional S/A, Tlalriepautla, Mexico.

*Francis L. Fennell* is now a Chemist in the Electrochemicals Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

*Maurice Finston* has been transferred from New York City to Philadelphia, Pa., as Purchasing Agent for the Philadelphia Quartermaster Depot.

*David B. Gearhart* is now Manager of the Kalamazoo, Mich., Office of the General Electric Co.

*J. B. Gough* has retired as Engineering Consultant for The Mead Corp., Chillicothe, Ohio, and will reside in Clearwater, Fla.

*Ralph S. Grant*, formerly of the Weyerhaeuser Timber Co., is now Assistant Pulp Mill Superintendent for the East Texas Pulp & Paper Co., Silsbee, Tex.

*Raymond A. Greene* of the Monsanto Chemical Co. has been transferred from Tuscaloosa, Ala., to New York City.

*George A. Hadlow, Jr.*, formerly Carthage, N. Y., Mill Superintendent of the St. Regis Paper Co., is now with the Guardian Paper Co., Oakland, Calif.

*Albert M. Hendricks* of the Hopper Paper Co. has been transferred from Reading Paper Co. to Taylorville, Ill., as Assistant Manager.

*Hideaki Hosaka* is now Chief of Research for the Forest Products Research Institute, Osahikawa, Japan.

*Alexander E. Hutton* of the Westfield River Paper Co. has been transferred from Russell, Mass., to West Conshohocken, Pa., as Laboratory Supervisor.

*A. Halvar Lundberg* is now President of A. H. Lundberg, Inc. and Lundberg Ahlei, Equipment, Ltd., Seattle, Wash.

*Lenart A. Lundberg* is now Secretary-Treasurer of same.

*Bruce W. Martin*, formerly of the American Box Board Co., is now Vice-President in Charge of Operations of Gardner Board & Carton Co., Middletown, Ohio.

*George H. McGregor*, formerly of the Minnesota & Ontario Paper Co., is now Manager of the Columbia River Paper Mills, Vancouver, Wash.

*Jaye A. McKenney*, formerly student of the University of Maine, is now a Technical Apprentice of the International Paper Co., Chisholm, Me.

*Alan A. Nyitray* is now Vice-President of Eastwood-Nealley Corp., Belleville, N. J.

*Robert C. Paris* is now in Charge of Quality Control for the Kalamazoo Vegetable Parchment Co., Kalamazoo, Mich.

*Robert W. Petrie*, formerly of the National Biscuit Co., is now Packaging Chemist for Thomas Lipton, Inc., Hoboken, N. J.

*Geoffrey Pyner*, formerly of the Whakatane Board Mills, Ltd., is now with the Tasmanian Board Mills Ltd., Lannceton, Tasmania.

*C. Lawrence Reese, Jr.*, formerly of the Whiting Paper Co., is now Technical Service Engineer for the Westfield River Paper Mills, Russell, Mass.

*Joseph S. Reichert*, Manager of Peroxygen Products Development for the du Pont Electrochemicals Dept., retired on September 1 and will reside in Lewiston, N. Y.

*Albert K. Roach*, formerly of William H. Welsh Co., is now Secretary-Treasurer of Donal, Inc., Cicero, Ill.

*William F. Scanlan* is now Sales Representative of the Whiting Corp., Ardmore, Pa.

*John G. Senese, Jr.*, formerly of the American Cyanamid Co., is now Project Engineer for the Westvaco Chlor-Alkali Div., South Charleston, W. Va.

*David B. Snedeker* is now President of Wm. A. Reed & Co., Lyons, Ill.

*John E. Spaulding* of the Marathon Corp. has been transferred from Oswego, N. Y., to Menasha, Wis., as Chief Paper Mill Superintendent.

*Martin E. Swinford*, formerly of Supples Hesse Corp., is now Chemist for Hill-Hentschel Ink Co., St. Louis, Mo.

*John E. Tasman*, formerly of the Columbia Cellulose Co. Ltd., is now Senior Chemist for the Pulp & Paper Research Institute of Canada, Montreal, P. Q.

*James S. Tipka*, formerly of the Marinette Paper Co., is now Felt and Wire Analyst in the Staff Technical Service Dept., Scott Paper Co., Chester, Pa.

*Richard Waack*, formerly student of the New York State College of Forestry, is now Chemist for the Dow Chemical Co., Midland, Mich.

*Albert L. Wiley* is now Assistant to the Production Manager of the Riegel Carolina Corp., Acme, N. C.

*Charles B. Williams*, formerly student of the New York State College of Forestry, is now in the research and development group of the Riegel Paper Corp., Milford, N. J.

*Clayton B. Willis*, formerly of the Industrial Chemical Specialties Co., is now the owner of Willis Timber Co., Gilmer Tex.

*Howard B. Wilson*, Southern District Sales Engineer for the Pusey & Jones Corp., is now located at 128 E. 66th St., Savannah, Ga.

*Edward A. Wodka* is now Technical Assistant Superintendent of the Paterson Parchment Paper Co., Bristol, Pa.

*Chien Jo Yeh* is now a graduate student in the Dept. of Chemical and Metallurgical Engineering of the University of Michigan, Ann Arbor, Mich.

*Robert N. Zabe*, formerly of the Champion Paper & Fibre Co., is now Assistant Professor of the Pulp and Paper Dept. Western Michigan College, Kalamazoo, Mich.

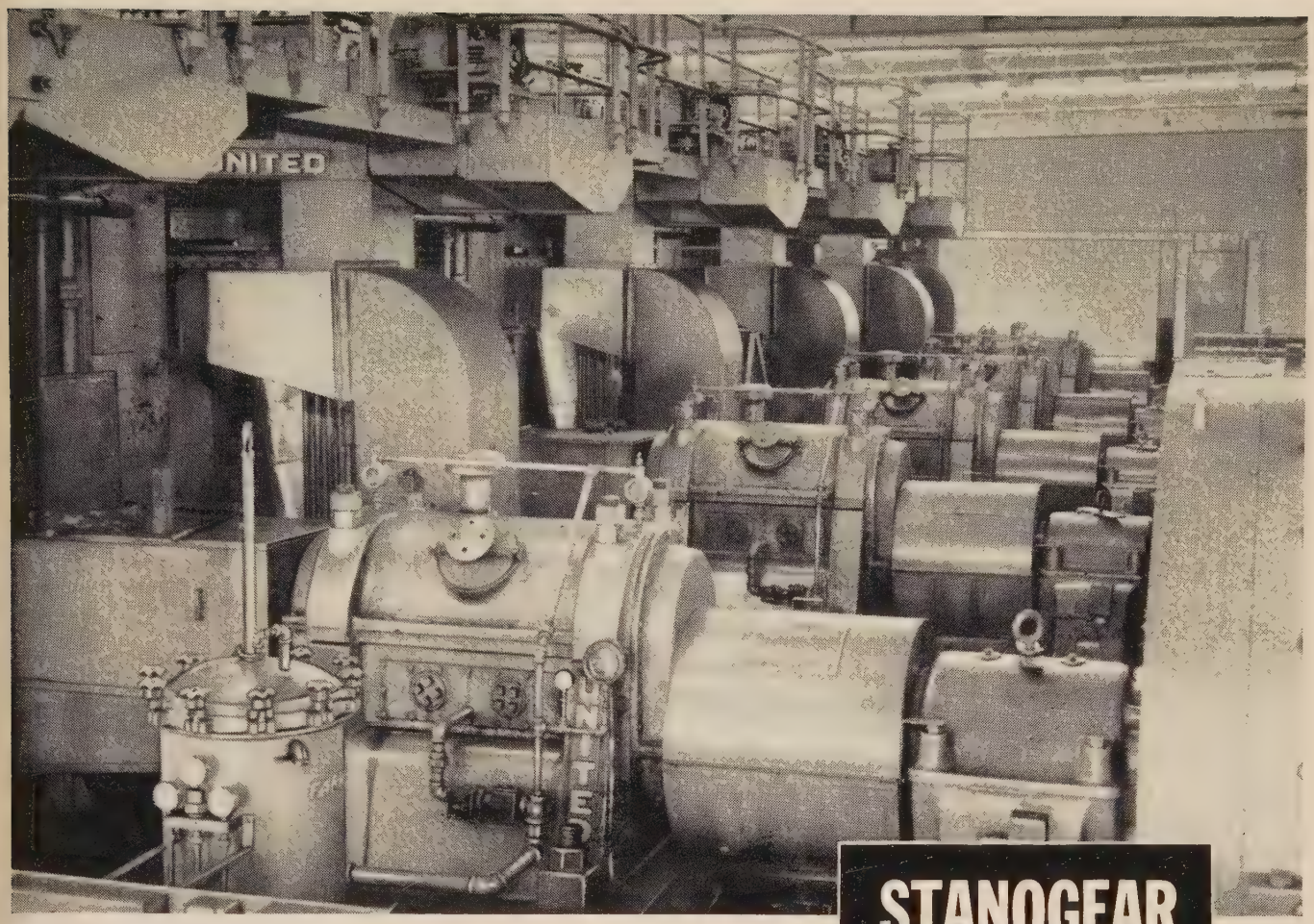
*Harold F. Zigmund*, formerly of the Mosinee Paper Mills Co., is now Assistant to the General Manager of the Blandin Paper Co., Grand Rapids, Minn.

*William R. Zwecker*, Packaging Consultant to Sunkist Growers, has been appointed to the consulting staff of the Stanford Research Institute, Palo Alto, Calif., for special research projects on packaging.

* * *

*Miss Ann Howard*, Librarian, has been appointed official corporate representative of The Mead Corp., Chillicothe, Ohio, in TAPPI.

* * *



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*Arthur C. Bird*

*Paul B. Corning*

*Roderick O'Donoghue* announced on September 1 that he has taken *Arthur C. Bird* and *Paul B. Corning* as partners in his established business of consulting and designing engineers to the pulp and paper industry. The partnership is to be known as Roderick O'Donoghue & Co., 420 Lexington Ave., New York 17, N. Y. Arthur C. Bird was 30 years with Hardy J. Ferguson and Mr. Corning has spent 28 years in the industry. Mr. O'Donoghue has had 42 years' experience in the pulp and paper industry.

*Robert Strasser*, Executive Vice-President, has succeeded *Norman H. Nuttal* as the official representative of Stein, Hall & Co., New York, in the Technical Association.

*Norman K. Janison*, Vice-President and Manager, has succeeded *Allen C. Huffman-Wolfe*, Southern Corp., Atlanta, Ga., in TAPPI.

* * *

The name of the Northern Regional Research Laboratory, U.S.D.A., Peoria, Ill. has been changed to Northern Regional Utilization Branch, U.S. Dept., of Agriculture.

## *Industry Notes*

PRODUCTION

Consumption of paper in the United States in the first half of 1954 was only slightly below that of the comparable period of last year, it was reported recently.

The total for the 1954 period was 15,420,000 tons or 1.8% less than the 15,701,000-ton total for the first half of 1953.

Eased demand for paperboard was the chief factor in the moderate decline in over-all consumption of paper. While production of other main types of paper, such as tissue and newsprint, was slightly ahead of last year, output of paperboard in the first half of 1954 was about 3.5% below that of the like 1953 period. Total output in the 1954 period was 6,010,000 tons.

Paperboard, which is used largely for shipping and other packaging, is a sensitive index to general business trends. Slackened demand for this commodity in the first half of the year was therefore a measure of the general business trend.

Paper and board production during June amounted to 2,269,912 tons. This compares with the 2,269,410 tons produced in June a year ago. Paper production, amounting to 967,359 tons, was a 3% increase over the 941,409 tons produced in June a year ago. The output of paperboard grades during this month, amounting to 1,033,042 tons, reflected a 3% decrease from the production of 1,063,111 tons reported during June, 1953.

*Newsprint.* Production and shipments of newsprint in North America during July, 1954, amounted to 600,303 tons and to 578,283 tons respectively, compared with corresponding

*Roderick O. Donoghue*

totals of 579,375 tons and 584,967 tons in the same month of 1953. North American production in July, 1954, exceeded that in any previous July. Output in the United States was 96,324 tons and shipments were 96,597 tons while Canadian (including Newfoundland) production amounted to 503,979 tons—the peak July record to date—and shipments totaled 481,686 tons.

Through the end of July this year United States mills turned out 33,954 tons or 5.4% more newsprint than in the first seven months of 1953 while output in Canada also was 129,600 tons or 3.9% greater than in the corresponding period of last year, thus the total continental increase amounted to 163,554 tons or 4.2%.

*Paper Bags.* Output of bag paper was 9.4% greater than in the first quarter of 1953. Unbleached kraft paper increased 9.7% and all other types increased 4.5%. January output of bag paper was heavy by comparison and 21.1% over the January, 1953, total; production tapered off in February and March, with March total 5.7% below the same month of last year. Compared with fourth quarter total of 243,306 tons, output of kraft bag in the first quarter dropped 4.7% and all other types dropped 27.6%. Total first quarter production of bag paper was 6.3% below the fourth quarter 1953 volume.

First quarter demands for paper bags were below the same period of 1953. In some areas decreases of 10 to 20% were reported. Similarly, bag operations were off from the fourth quarter volume. Although activity was slow in January and February, business picked up in March and continued into April. Order backlogs were below "normal." Bag users inventories were down. Buying was slow with most purchases made on an immediate delivery basis. Paper costs were low and resulted in a slight reduction of bag prices. Raw material inventories were light. Business for the balance of the year is expected to be satisfactory.

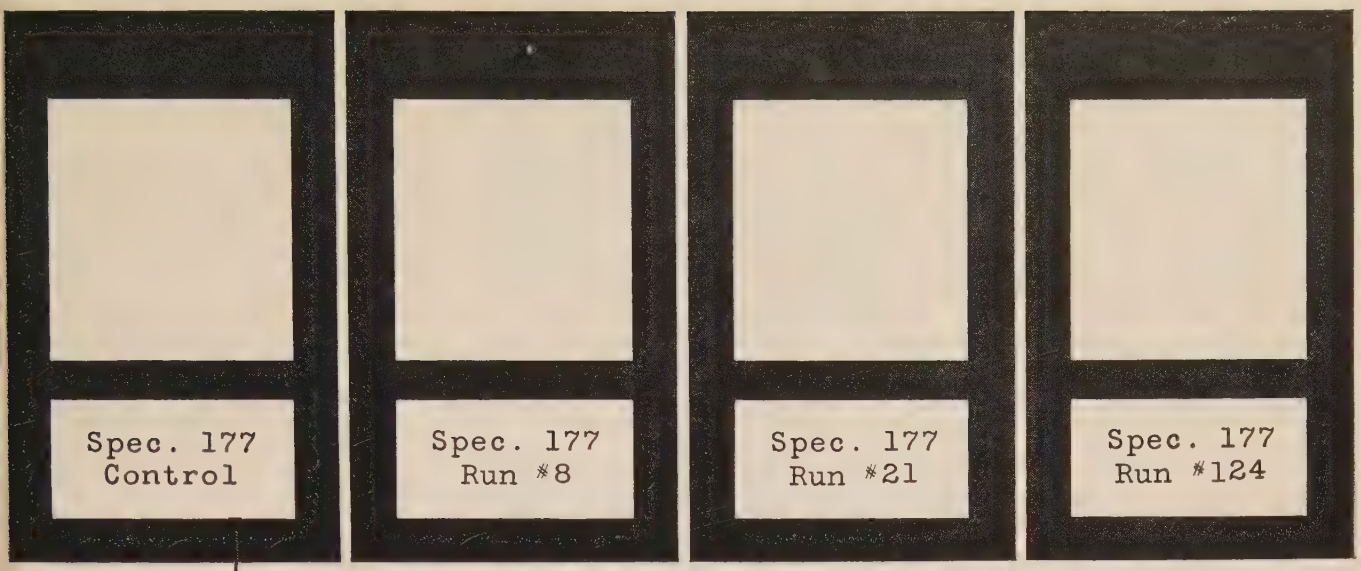
*Folding Paper Boxes.* Production of folding boxboard in the first quarter was 14% below the comparable period of 1953. Although January volume exceeded January, 1953, by 2.4%, output of both February and March was lower. Compared with the fourth quarter output of 594,477 tons, first quarter production increased 9720 tons or 1.6%.

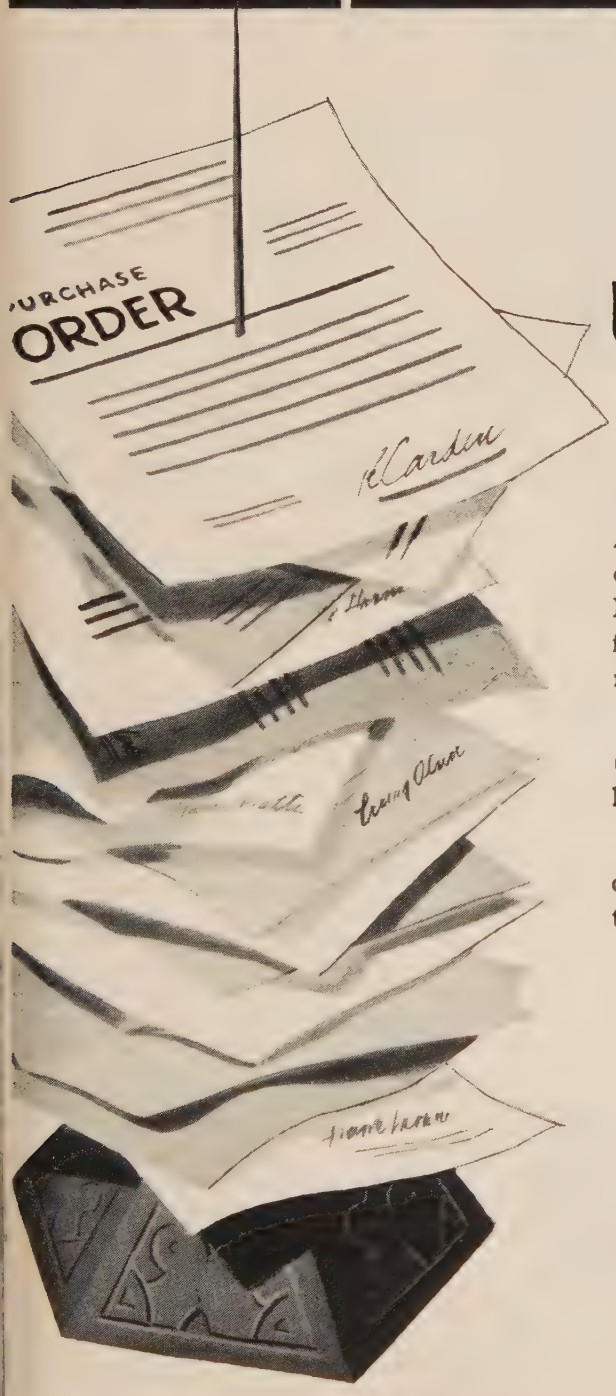
Folding paper box operations in the first quarter were maintained at approximately the first quarter 1953 level. Shipment for the period as measured by dollar volume was 2.1% above the 1953 total. Shipments billed in terms of tonnage were down 0.3%. Orders entered for the first quarter were only 0.1% less than the dollar volume of same period of 1953; tonnage of orders entered was 3.0% less.

Although box operations started slowly in January a pickup occurred in February and held fairly steady through March. Box shipments in January were 4.8% below the tonnage shipped in the same month of 1953. Dollar volume for January shipments was also off 2.8%. Orders entered for the same month were 7.7% below in tonnage and 6.2% below in dollar volume than January, 1953, totals. February demands were heavier with tonnage shipped 3.1% above the 1953 total; similarly, dollar volume of shipments billed was up 6.7%.

*Setup Paper Boxes.* Production of setup boxboard in the first quarter was 3.4% below the first quarter 1953 total and 3.2% above the 177,663 tons of the fourth quarter of 1953.

Demand for folding paper boxes during the first quarter was from 10 to 20% below the heavy 1953 level. Similarly, a seasonal decrease also resulted in box operations dropping below the fourth quarter total. Order positions were below the first

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quarter 1953 volume with backlogs whittled down to one to two weeks. Container users ordered smaller quantities and generally insisted on immediate delivery. Forward buying was low. Box inventories were also conservatively low. Raw materials were plentiful with prices steady. Inventories of raw materials were held at a minimum level. Box prices were steady to lower. By the end of the quarter business had picked up as advanced orders in many areas were placed earlier than usual. Boxmakers view the near future business in terms of "normalcy."

*Corrugated and Solid Fiber Shipping Containers.* First quarter production of container board was 6.5% below the corresponding period of 1953. Output of liners was off 5.7%, corrugating materials was off 6.8% and chipboard was off 14.9%. With the exception of a 2.1% increase in the output of chipboard in March, monthly production of all other categories was below the corresponding months of 1953.

*Fiber Cans and Tubes.* Fiber can and tube business was slow during the early part of the first quarter but picked substantially in March. As a result, sales for the period were approximately 13% below the heavy first quarter 1953 volume. Can sales were about 10% lower with sales of spiral and convolute tubes and miscellaneous specialty items off slightly more. In part, the sales decrease resulted from the stretch-out or termination of defense contracts on ammunition containers. Civilian demand for cans and tubes for the first quarter was about 5% below the corresponding period of 1953. First quarter demands for containers for frozen foods and biscuit dough were heavier than in previous periods.

First quarter operations were also approximately 9% below the fourth quarter 1953 volume. Although can sales were off about 14%, sales of spiral tubes and miscellaneous specialties were approximately 1% lower. Convolute tube shipments were down about 5% from the first quarter 1953 level.

Container prices were slightly lower. Raw material costs declined "very little." Inventories were normal. Business outlook is "good" for the first six months.

### Wood Pulp

Total wood pulp imports for January–June, 1954, in comparison with January–June, 1953, are as follows:

| | *1954*[a] | *1953*[a] | *Per cent change* |
|---|---|---|---|
| Canada | 821,644 | 793,908 | +3 |
| Sweden | 102,111 | 184,163 | −45 |
| Finland | 59,934 | 86,867 | −31 |
| Norway | 8,049 | 22,895 | −65 |
| Germany | ... | 588 | ... |
| France | 50 | 75 | −33 |
| Austria | ... | 6,164 | ... |
| Portugal | 25 | ... | ... |
| Total Europe | 170,169 | 300,752 | −43 |
| Total World | 991,982 | 1,094,660 | −9 |

[a] Short tons.

### Wastepaper

The building materials mills of the United States last year consumed something like 800,000 tons of wastepaper in the manufacture of 1,312,972 tons of building papers and 1,375,980 tons of building boards. During the first half of this year, the manufacture of both building paper and building board has been below the first six months of 1953, and consequently wastepaper consumption by these manufacturers has been less than last year. However, with the signing of the Administration's new housing act by President Eisenhower, the construction industry looks for a building boom during the coming months, and the manufacturers of building materials, namely building papers, boards and roofing, expect increased demands for their products. This would result in increased demands for the bulky grades of wastepaper used in their manufacture.

During the first five months of this year, the building materials industries consumed an average of 61,000 tons of wastepaper a month; in the comparable period of last year, the average monthly tonnage of wastepaper consumed was 64,000.

Consumption of wastepaper by the paper mills during the first half of 1954 increased by a few thousand tons over the first six months of 1953, according to reports of the Bureau of Census, while there was a drop in the consumption by other consumers. The paper mills, in the six months ending June 30, consumed 576,656 tons of wastepaper as against 571,154 tons consumed in the corresponding half year of 1953.

Paperboard mill consumption in the first six months of this year amounted to 3,046,325 tons as against 3,352,254 tons consumed in the first half of last year. Other consumers, including the wet machine board mills, the roofing, building board, and building paper mills, consumed 417,375 tons in the first six months of 1954 as against 433,906 tons in the like period of 1953.

In the first half of the year, the paperboard mills consumed 75.4% of the total consumption, the paper mills 14.2%, and the various other mills 10.4%.

### Latin America

The development problems of the Latin American pulp and paper industry are being studied and discussed by experts from various parts of the world, who are meeting in Buenos Aires from October 18 to November 2 of this year. This meeting is being sponsored by the Economic Commission for Latin America, the Food and Agriculture Organization, and the Technical Assistance Administration of the United Nations, as well as by the Argentine government.

The principal pulp and paper experts and representatives of specialized institutions in Australia, Canada, France, Germany, Great Britain, Sweden, and the United States will be present or will send documents of a technical nature, which, together with those of the United Nations organizations and Latin American experts, will serve as a basis for an interchange of knowledge and experience and discussion of the problems hindering the development of the Latin American pulp and paper industry. The products of this industry are considered of vital importance for the economy and culture of the region.

#### *The Problem Before the Experts*

Latin America has an average annual consumption of 1,500,000 metric tons of paper, of which approximately 400,000 tons are newsprint which is only manufactured in four countries—Argentina, Brazil, Chile, and Mexico—and in quantities which do not exceed 24% of their own total consumption. Although during the last 15 years the domestic output of paper in Latin America has been doubled, the region imports more than two fifths of the paper and cardboard it consumes, and almost half the pulp required by the paper industry. This means that about 70% of Latin America's pulp supplies are dependent on imports from other regions of the world, particularly Europe and North America (United States and Canada). Such dependence on imports has resulted in a slower increase in Latin American paper consumption than was to be expected from the rate of growth of its per capita income. The great shortage of foreign exchange which, in general, has obliged the Latin American countries to restrict their imports, is partly responsible for this situation.

With regard to the future, a disturbing element is the fact that an adequate increase in Latin American pulp and paper consumption would represent a demand that probably could not be satisfied by the traditional world suppliers of these commodities, especially considering that these countries have also to face a large increase in their own consumption, utilizing forestry resources which are gradually reaching the maximum limits of their economic yield. Furthermore, the

studies carried out by ECLA and FAO indicate that even though the present imports of pulp and paper are maintained and all the mills at present under project are constructed and put into operation, there would still be a considerable deficit in supply, which would inevitably result in the paper supply being less than the quantities required for the cultural and economic progress of the region.

Therefore, the problem is establishing the necessary productive capacity to absorb the normal growth in the demand of the region. As the development of the Latin American paper industry would have to be based on short-fibered raw materials, an increase in the regional productive capacity would stimulate rather than discourage imports of long-fibered products from Europe and North America. This, however, would establish a rational proportion between imports and production and would mean the disappearance of the strict dependency on foreign imports that hinders increased consumption, as has occurred formerly.

*Agenda for the Meeting of Experts*

It is of concern to the United Nations to ensure that future development programs are based on a better knowledge of probable paper consumption, and also that measures should be adopted to ensure an appropriate paper supply, consistent with the increase in per capita income of the Latin American countries that is expected to occur in the immediate future.

It is for this reason that, in the studies being carried out by the ECLA Secretariat in cooperation with UNESCO and the Economic Commission for Europe, an effort will be made to establish the figures on which development should be based, considering not only the possibilities of creating a greater Latin American productive capacity, but also the possibility of counting in the future on the necessary pulp products from Europe and North America.

Over 95% of Latin America's forest area is composed of tropical and subtropical forests, and, in consequence, long-term industrial and paper development programs must necessarily concentrate on the utilization of these abundant resources. Until recently, technical knowledge relating to the manufacture of wood pulp was based on the utilization of conifers and some temperate hardwoods, which are most plentiful in the traditional paper-supplying countries. The need to expand the sources of raw materials has already induced certain countries to study, with a fair amount of success, the utilization of tropical species. France has established a pilot plant in Abidjan, in French West Africa. The Forest Prod-


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ucts Laboratory of the United States Department of Agriculture has devoted special attention to testing tropical species and has developed methods for their utilization. In Latin America, little has been done in this field up to the present, and there is insufficient industrial experience on which to base a study of the economic aspects of the exploitation of tropical forests for paper and pulp production.

With this object in view, ECLA and FAO experts are carrying out preliminary studies in Yucatan (Mexico) and Amapa (Brazil), which will be used as a basis for discussion at the meeting of experts. These studies consist of preliminary forestry surveying, pulping tests of regional species, projects of wood extraction and transportation, and preliminary plans for pulp and paper mills.

Other subjects of great interest to Latin America to be discussed during the meeting of experts, will be the influence of the size of mills on investment and production costs; the influence of the integration of paper mills with those of pulp: the possibility of producing newsprint from tropical hardwoods, and the experience gained in other countries in the utilization of hardwoods in the production of pulp and paper.

### U.K.

Canada is the chief source of supply for newsprint but Sweden, Norway, and Finland provide the bulk of other paper products for the United Kingdom. The United States supplied paper-related products, and manufactures valued at $6,242,321 in 1953—approximately 12% of the total imports of these products. Liner container board accounted for $5,828,562 while kraft, special industrial, absorbent, and thin fine paper constituted the major portion of the remainder of the imports from the United States.

### Chile

Chile is to erect a 75,000-ton kraft pulp mill, valued at $25,000,000. Financing will be done on a long-term credit basis, secured through two business firms in Cologne and Hamburg, Germany, which firms will supply most of the machinery and supervise construction.

### U.S.S.R.

L. P. Zherebov, writing in the Soviet paper industry journal, states that while it is perfectly true that one third of the Soviet Union is covered by forests, timber is a comparatively costly and uneconomic source of cellulose for paper production.

There is a large virtually untapped source of cellulose that ought to be properly exploited. The source is straw. Soviet industry is able to produce all types of paper from straw. In 1950, 200 million metric tons of straw were potentially available in the Soviet Union; at a pulp extraction rate of 40%, they would yield a total of 80 million tons of cellulose.

A student of Soviet economic affairs in London commenting on Zherebov's article, says that apparently Russia intends to offer in the very near future appreciable quantities of timber and wood pulp to the West, regardless of deterioration in quality of domestic paper supplies.

### Gaza

Barren sand dunes compose almost half of the 175 square miles of the Gaza strip, a narrow fringe of land between Israel and the Mediterranean in which live more than 280,000 people—over 200,000 Palestinian refugees and 80,000 original inhabitants.

At first these dunes presented a barren prospect to the UN Relief and Works Agency in its efforts to provide morale-building and gainful employment for the refugees in its charge, 70% of whom are farmers.

An ingenious answer to the problem was found, however, in the mimosa tree, which was used many years ago to "anchor" a number of areas in the Gaza vicinity. Mimosa, known also as Australian acacia, or wattle, is a quick-growing, bushy tree which, in as little as three years, can stabilize shifting sands by covering them with waving green thickets. Almost immediately, grasses and other low vegetation will begin to sprout.

This system of mimosa planting was widely used in Gaza before the outbreak of the Palestine conflict. Recently, the idea was revived by UNRWA planners who embarked on a scheme to anchor the Gaza strip's drifting sands and make at least one-third (3750 acres) of the clay plain beneath them available for cultivation by the refugee farmers.

In cooperation with the Egyptian Administration, UNRWA crews have already set out a million potted mimosa seedlings in government nurseries. Next February and March, when rain-soaked sands begin to warm, and winter winds lose their bite, the seedlings will be spread two meters apart over 1650 acres of the Gaza dunes. During the next two years, another two million seedlings will be set out.

### Georgia

Georgia led the entire South in pulpwood production in 1953. Citing latest figures compiled by the U. S. Department of Agriculture, Nelson M. Shipp, secretary of the Georgia Dept. of Commerce, said that of the South's total output of 16,127,000 cords, Georgia produced 2,879,168 cords. Of these 2,748,853 cords were pine, 124,613 were hardwood, and 5702 were chestnut.

The South as a whole produced 61% of the pulpwood harvested in the nation. Georgia's 1953 figures showed a gain of 366,000 cords over 1952.

### Wisconsin

A twelve-man team of chemists, biologists, and engineers from four major research centers of the pulp and paper industry recently made an intensive survey along several miles of the Wisconsin River below two big sulphite mills. The study was both chemical and biological.

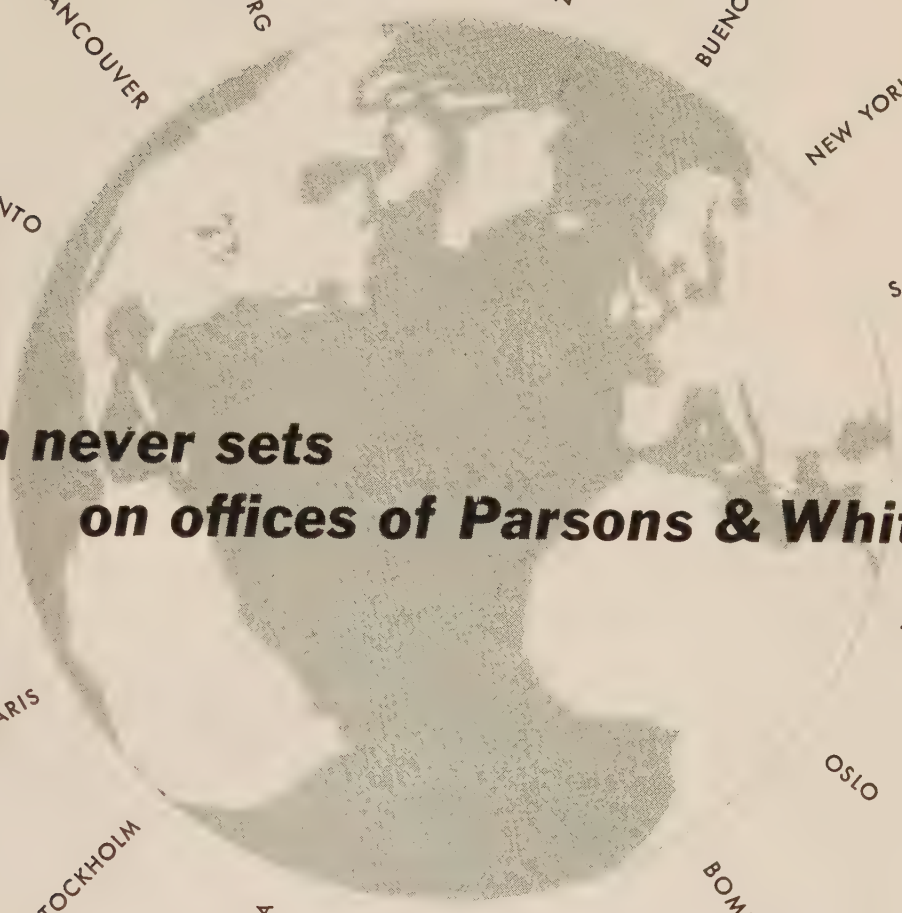
The scientists dipped samples of stream water at regular intervals along a 50 to 60-mile stretch of river from Biron to Castle Rock. They dredged up specimens of marine animal life off the bottom. Also they poked into fibrous sludge beds left in the river from bygone decades.

Top men of the expedition are G. K. Dickerman, technical director of Consolidated Water Power & Paper Co., Wisconsin Rapids; Truman A. Pascoe, research director of Nekoosa-Edwards Paper Co., Port Edwards; and Averill J. Wiley, technical director, Sulphite Pulp Manufacturers' Research League, Appleton. They directed a group of chemists and engineers from their own technical staffs. Willis Van Horn and R. F. Balch of The Institute of Paper Chemistry, Appleton, planned and carried out the biology phase of the survey. An engineer of the state Committee on Water Pollution was present by invitation as an observer.

The three directors list three major purposes of the study. They want a scientific yardstick for measuring effectiveness of pollution abatement steps taken by their mills. They seek to supplement data obtained by the state in order to understand the river's condition more clearly. They also hope to throw more light on a theory recently advanced by university biologists that sulphite liquor sugars in surface waters supply food that starts the water life cycle. In this cycle, microscopic organisms feed on the sugar and larger organisms feed on the smaller ones, leading eventually to plentiful food for game fish. The survey party is out to learn whether this accounts for the bigger fish and better fishing found in Wisconsin streams and lakes beyond the pollution zones below some sulphite mills.

### Hardboard Hearings

The U. S. Tariff Commission is making an investigation of the domestic hardboard industry and of the effect of the present tariff status of imported hardboard upon the domestic

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industry. Public hearings of interested parties will be held in Washington beginning Dec. 7, 1954.

### Employment of Minors

A hazardous occupation order issued recently by the U. S. Dept. of Labor prohibits boys and girls under 18 years of age from operating or assisting in operating 14 different types of power-driven paper products machines, including: arm-type wire stitcher or stapler, circular or band saw, corner cutter or mitering machine, corrugating and single- or double-facing machine, envelope diecutting press, guillotine paper cutter or shear, horizontal bar scorer, laminating or combining machine, sheeting machine, scrap-paper baler, or vertical slotter. Also included is the occupation of operating or assisting to operate any platen die-cutting press, platen printing press, or punch press which involves hand feeding of the machine.

### Forestry Papers

The following publications have been issued by the Northeastern Forest Experiment Station, Upper Darby, Pa.:

*Forest Research Notes* No. 32, Surface Sterilization of Hybrid Poplar Cuttings; No. 33, Effect of Sterilizing Treatments on Survival and Growth of Hybrid Poplar Cuttings; No. 34, Do Thinning and Prescribed Burning Affect the Growth of Shortleaf Pine?; No. 35, Mice Cause Severe Damage to Virginia Pine Reproduction; No. 37, A Potential Timber-Type Chinese Chestnut.

*Station Paper* No. 70, Factors Affecting Wind Damage in Selectively Cut Stands of Spruce and Fir in Maine and Northern New Hampshire.

*Forest Statistics Series*, New York, Nos. 4, 5, and 6, Forest Statistics for New York.

### AFA Action Program

The American Forestry Association has drawn up a Proposed Program for American Forestry, stating the policies which the AFA recommends for public, legislative, and administrative action and for support by industries, civic organizations, and public-spirited citizens.

### Forest Fire Insurance

Whether the husbandry of our forest and timber resources is good or bad depends largely on the decisions of private owners. In New England private owners hold 95% of the commercial forest land and the same percentage of the saw timber. Nearly 60% of the saw timber and three quarters of the commercial forest land in the United States are privately owned.

Almost four and a quarter million private owners hold an average of 81 acres to make up a total of more than 340 million acres of commercial forest land in the United States. The aggregate value of the timber and wood as it stands on these properties can be conservatively estimated at more than $10 billion. Although the value of the average individual property is only about $2500, some single ownerships run into the millions.

The 29 million acres of privately owned commercial forest land in New England is divided among almost 244,000 separate holdings with an average size of 120 acres. There are only about 200 ownerships of more than 5000 acres each, of which 45 are in excess of 50,000 acres, but these larger holdings embrace 40% of the forested area. The Great Northern Paper Co. and the Pingree Estate, in Maine, are examples of the more extensive ownerships. The value of the standing timber and wood on New England's commercial forest area has been estimated to be in excess of $500 million. The average value per property is only about $2000; but if the value distribution is in proportion to the area distribution, the 200 larger ownerships have an average value of more than one million dollars. Some are worth much more than this and it is obvious that the number of individual properties with values many times the average must be substantial. A similar situation exists in the nation as a whole.

It is reasonable to expect the owners of these forest properties to undertake programs of modern improved forest management—the growing of timber as a commercial enterprise—only when they can do so with a justifiable expectation that the financial return will be in line with the benefits they can obtain from other uses of capital, management, and labor, with full consideration of the involved risks. Some of the risks incurred by private forest enterprise—particularly excessive taxation—have been discussed in previous issues of the *Monthly Review*.* The purpose of this article is to take a look at the physical risks that forestry enterprise faces, particularly the fire risk, and the possibility of abating this risk through application of the insurance principle.

Forestry enterprise differs, in this physical risk aspect, from practically all other private business undertakings, Insurance against possible loss from the common perils, particularly fire, is a widespread practice in most businesses. It is extremely rare in the forestry business. It is not necessary to look far for an explanation of this phenomenon.

A superabundance of virgin forests, from which lumber and wood supplies could be had practically for the taking, surrounded the early settlers. Little thought was given to forest preservation or the future supply of forest products. If fire ran wild in the woodlands, nobody cared particularly. This attitude toward the forests began to change during the last quarter of the 19th Century as the realization dawned that they might not, after all, be inexhaustible.

#### *Protection Cuts Forest Fire Losses*

The forest fire protection effort began in a small way about 50 years ago. Since then it has spread over all but a small part of the nation. Nearly a quarter of a million dollars was expended in 1912 to provide protection on an area of 61 million acres of forest land owned privately or by the states. By 1952 the protected area had increased to nearly 369 million acres and the annual financial outlay by individuals and state and federal governments amounted to nearly $36 million, not quite 10 cents per acre protected. In addition, local governments and private owners and operators made capital outlays for forest fire equipment and improvements totaling more than $16 million

How effective is forest fire protection in New England and the nation? Is it really getting results from its efforts to curb the ravages of forest fires? Substantial gains have been made: all of the private and state-owned forest areas of 31 states, mainly in the North and West, were under organized protection in 1952. In 13 states, mainly in the South, areas protected ran between 34 and 92% of the total. Data are incomplete in four states where little privately owned forest land exists. During the 10-year period 1943 to 1952, the average area burned annually on these protected lands varied between a low of 0.01% in Wyoming and a high of 2.57% in Florida.

Although both the best and poorest records were established in states where fire protection is incomplete, this evidence is not conclusive, particularly with respect to Wyoming's excellent record. The effectiveness of the protection is an important factor but other influences, favorable or unfavorable, take effect. The combination of these other effects is favorable to small fire damage in Wyoming; Florida is among the states where they are at their worst. Looking at all the states in combination provides more readily credible evidence.

During the observed 10-year period, the average area burned annually was 0.47% of the total area protected in the 31 states where full protection was provided. In the 13 states where only partial protection was afforded, the average area burned, on the protected portion, was 1.65%, three and one half times as great. The purpose of these comments is merely

* *Monthly Review*, June and August, 1949; February, 1952.

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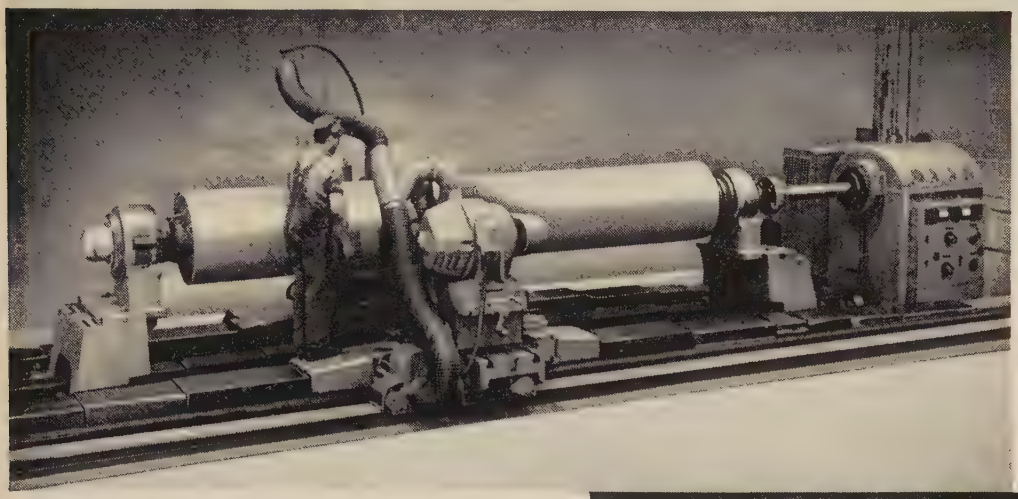

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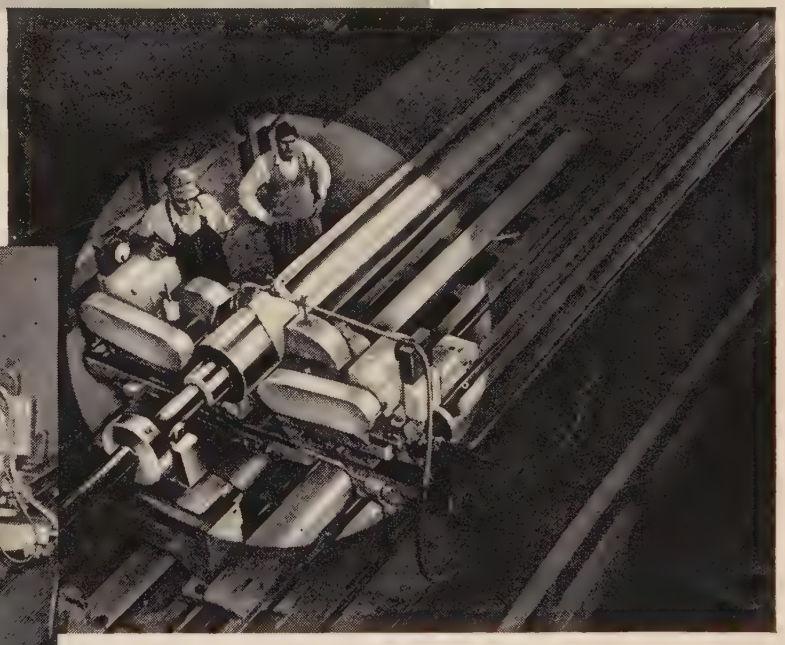

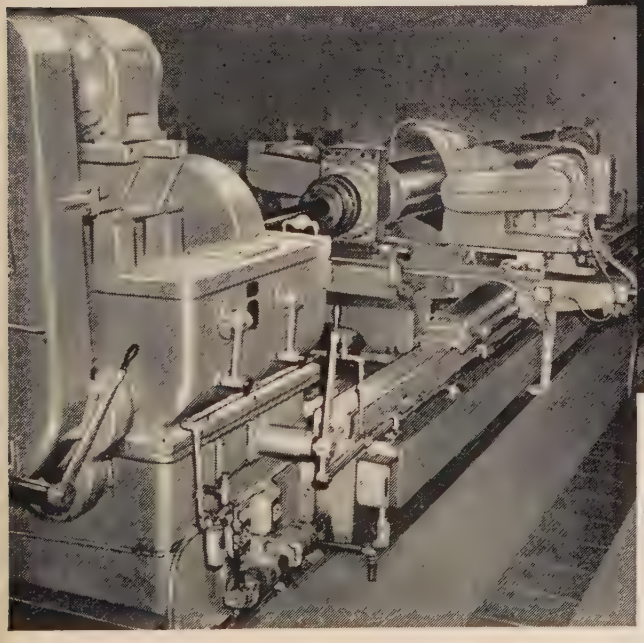

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1954

to show the extent to which the forest fire menace is being held down in a large portion of the United States.

The second chart shows that organized forest fire protection is gradually getting better results. The average area burned annually on protected state and private lands was 1.1% during the latest 24 years for which data are available—1929 to 1952. During the first eight years of this period the annual average was 1.5%. During the second eight years it dropped to 1.0% and, for the last eight years of the 24-year period, to 0.8%—only slightly more than half the percentage burned during the first eight years.

The area burned in an average year provides an important indication of the long-term trend of forest fire damage. But it is an unreliable indicator, in any one year, of the damage ratio in financial terms. An exhaustive study of forest fire losses, conducted some years ago,† showed that the ratio of financial loss to total forest property values is a lower figure than the ratio of area burned to total area. Forest fires do not consume living trees. They often kill them, but the retention of salvable value is a general rule. Some fires burn lightly and cause slight or even no damage to timber of marketable size. The recorded area burned frequently includes recently-cut land or land bearing only worthless scrub and brush. On the other hand, forest fires are sometimes "crown" fires that kill all the trees they touch. If the killed timber is below usable size, all the value it possesses is lost. Salvage is not possible as is the case with killed timber of marketable size. Salvage was an important factor, for example, after the Tillamook conflagration of 1933 in Oregon and the conflagration in Maine in 1947. In both cases the net loss was substantially below the gross figure indicated by the quantity of timber killed.

*Measurement of Financial Loss*

It is possible, by combining the statistics from a large number of burned areas, to derive averages which indicate the general relationship between area burned and financial loss. The 1939 study of forest fire losses in the northeastern states showed that average annual losses were $775,000 on a total value at risk of $573 million. This is 0.13%—13 cents per $100 of value. The ratio of area burned to total area during the same period was 0.42%. These area figures correspond to those presented in the charts which show a ratio of area burned in the United States between 1929 and 1952 of 1.1% and, between 1943 and 1952, of 0.8%.

During the most recently recorded 10-year period, the loss in value on the protected state and private lands in the nation was 0.25%—25 cents per $100 of total value exposed to fire. These are national figures including the states in the South. In many individual states the annual financial loss expectation based on the record is much less than this. For example, it is one cent per $100 in Wisconsin, a heavily forested state. In Washington and New York it is four cents. In New England as a whole the financial loss expectation is five cents for each $100 of forest land. At the top extreme, if the principle holds true, it is 80 cents in Florida. All of these figures are state-wide figures. Significant and measurable variations occur within the states.

The 1939 bulletin mentioned above reports on a detailed town-by-town study of Massachusetts. That state had an average annual area burned, during the 1943–52 decade, of 0.28%. Its value loss expectancy is thus nine cents per $100 per year. The 1939 study revealed the fact that forest fires tend to form a consistent pattern over the years and that towns in which loss expectancies are high can be quite readily identified. These are the towns which, according to the record, have more fires, or larger ones, or both, than do the other towns. The greater portion of the area burned during a period of years tends to be concentrated on Cape Cod and in 32 other towns. In the other 319 forested towns, the forest area burned annually falls far below the state average. This finding is significant because it indicates that, in the nation as a whole, forest fire loss probabilities on extensive areas are lower than the state averages indicate.

How does the forest fire loss probability affect the prospects of the application of the insurance principle to forest fire losses in the nation? The above figures provide a basis for dividing the country into two general zones—one where forest fire losses are relatively low and one where they are relatively high. Broadly speaking, losses are low in the North and West and high in the South. This makes it more logical to look at the North and West for opportunities for initial application of the insurance principle. If aggregate losses are low, insurance premium rates can be low. Low premium rates favor prospects of success. If they are high and appear likely to absorb an excessive proportion of gross earnings, the owners will tend to avoid buying insurance.

The analysis shows, though, that there is, in the North and West, an aggregate forest value of nearly six billion dollars on almost 200 million acres under full, organized forest fire protection. The average over-all loss expectancy is 0.26% per year on the area basis—eight cents per $100 of value. With the probability that loss expectancies are less than these on many individual properties, the possibility of offering insurance at prices the owners would be willing to pay seems great. Furthermore, the aggregate value and geographical spread appear sufficient to fulfill the needs of practical and sound insurance underwriting.

Why, if underwriting conditions are as favorable as they appear, is the insurance principle not being more widely applied to forest fire loss? History provides a possible answer. Attitudes and practices of insurance organizations and forest owners continue to be colored by the memory of many years of unfortunate experience. The antiforest fire public education program may have some adverse influence.

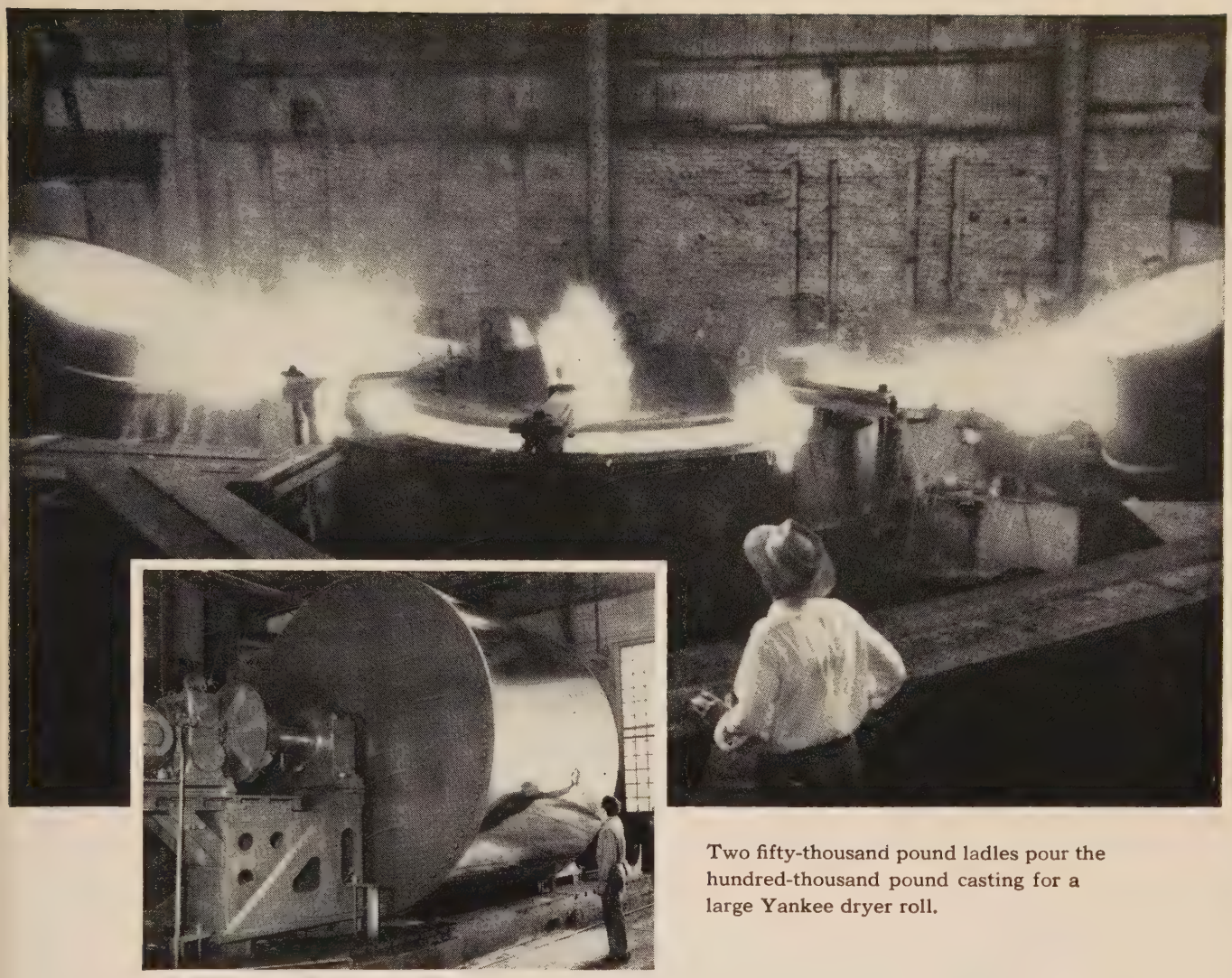
Forest fire insurance has been a subject of discussion and study for more than 30 years but, so far, no insurance organization has made a vigorous effort to develop the business as more than what they call an "accommodation" line. Forest owners have largely reconciled themselves to its absence, perhaps because of the impression that premium rates would be too high anyway.

The figures nevertheless provide evidence that an opportunity exists for a new pioneer undertaking in the business of forest fire insurance. Such a business, wisely and effectively undertaken, should find many thousands of prospective customers among timber owners engaging in forestry enterprise. Elimination or reduction of the fire risk would bring them positive benefits.

Some fire insurance company executives have expressed doubt that an effective demand for forest fire insurance exists. It may very likely be true that demand will remain substantially ineffective until premium rates are brought down to levels proportionate with prospective earnings. More importantly, it may be that the owners have reasonably accurate knowledge of what their fire risks really are and that they will not buy insurance until the cost becomes a close measure of them.

Designing and promoting a system of widespread forest fire insurance will not be a simple undertaking. There must be differential rating schedules by which a specific rate is tailored to the individual property being insured. Flat, or average, rates will not work because they are attractive only to the owners of properties where the risk is higher than the average. Use of them would soon bankrupt the insurance carrier. Premium rates must allow for possible inaccuracies in the data and the interpretation of them and for adverse selection of risks—the insured portion may not be a perfect sample of the whole. They must allow for accumulation of reserves against conflagrations, other contingent hazards, and the unknown factors.

† Forest Fire Insurance in the Pacific Coast States and Forest Fire Insurance in the Northeastern States. U. S. Dept. of Agriculture Technical Bulletins 551 and 651, 1937 and 1939, Washington, D. C.



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*Underwriting Procedures*

Policy forms must include the essential protective clauses—protecting both the insurer and the insured. There must be agreement on coinsurance and deductible requirements. Full indemnity may not be advisable, particularly at the start. Provision should be made for types of policies attractive to large owners who are self-insurers against ordinary fires.

A standard system for grading protection organizations would be indispensable following the general pattern by which cities and towns are graded according to the effectiveness of their fire and police departments, their water supplies, hydrant systems, and other protective facilities.

The insurance of forest properties against fire damage might well be patterned after the practices applied to the insurance of buildings and stocks of goods. Accurate indemnification of the insured's real financial loss, in compliance with the terms of the policy, should be the objective. There do not appear to be any insurmountable obstacles in the way toward this objective.

The evidence indicates that forest fire insurance offers the established fire insurance organizations, stock and mutual, an opportunity for business expansions in the United States. Fire insurance, whether provided by the established organizations or by new carriers formed by the forest owners themselves, has the capacity of conferring positive benefits on the business of growing timber and wood for the market.

Fire insurance would not eliminate all the physical risks that timber growing incurs. The risk of loss from tree disease and insect epidemics, wind, and other agents of destruction would remain. Systems of insurance against these risks might follow the firm and successful establishment of insurance against fire.

—from *Monthly Review*, Federal Reserve Bank of Boston, June, 1954.

## Newsprint

1. *Newsprint Use.* In the face of limited availability of the raw materials normally used for newsprint manufacture, the demand for newsprint in the United States, which has increased steadily for more than a decade, continues to rise. Approximately 6.1 million tons of newsprint, equivalent to 78 lb. per capita, were consumed in the United States in 1953, chiefly for newspaper use. A recent estimate of the American Newspaper Publishers Association places newsprint demand in 1960 at about 7.5 million tons, while the President's Materials Policy Commission has estimated that by 1975 requirements for newsprint will approximate 8.7 million tons.

For more than a quarter of a century, the bulk of the newsprint used in the United States has been supplied by Canada. The heavy concentration of newsprint production in Canada has been based upon conditions of competitive economics.

2. *Hardwood Potentialities.* Hardwoods hold out the possibility of major additions to the supply of newsprint pulping materials within the United States. As reported by the Forest Service in latest available data, annual growth of hardwood timber for the country as a whole was estimated to be about equal to that of softwoods. Hardwood growth was estimated to exceed hardwood drain by about 20%, whereas it was estimated that the softwoods were being overcut by a similar margin.

Hardwoods are being used increasingly for various grades of paper other than newsprint, but their utilization for the manufacture of newsprint in the United States has heretofore been of minor consequence. Recent technological developments, however, have shown the way to efficient and economic pulping of certain abundant hardwood species—primarily poplar, birch, beech, and maple—for use in making newsprint.

Industrial interest has already been demonstrated in the United States and elsewhere in growing and using hardwoods for newsprint, and important commercial beginnings have been made. Substantial supplies of suitable native hardwoods are economically available in the United States, and these supplies could be expanded if necessary to meet increased demands.

*Hardwood Pulping for Newsprint*

3. *Increased Hardwood Use.* Great strides have been made in recent years in the development of pulping technologies particularly suited to the processing of hardwood species on an efficient and economic basis. In 1953, about 4.4 million cords of hardwoods were used for the manufacture of pulp and paper in the United States, or about 15% of the total pulpwood consumed by the industry. This is more than twice the amount of hardwood pulpwood used a dozen years ago.

4. *Pulping Processes.* Certain technologies suitable for pulping hardwoods for newsprint use on a substantial scale have been developed and are in various stages of commercial realization. The most promising of these technologies are the chemigroundwood, neutral sulphite semichemical, and cold soda processes in addition to the conventional groundwood process.

Of the hardwood species, only poplar is suitable for groundwood pulping from the standpoint of general availability and of brightness, strength, and other qualities required for newsprint use.

Chemigroundwood, semichemical, and cold soda pulps made from poplar and certain other hardwoods have intermediate strength properties between softwood groundwood and chemical pulps, and also have other favorable properties which in blends of various proportions with poplar groundwood permit substantial displacement of the softwood chemical pulp as well as of the softwood groundwood normally used.

5. *Chemigroundwood Process.* The chemigroundwood process, which is commercially the furthest advanced of the newer hardwood pulping technologies for manufacturing newsprint, is suitable for pulping poplar, birch, beech, and maple (and possibly the gums) for newsprint use without bleaching.

This process, developed at the New York State College of Forestry, Syracuse, N. Y., has been adopted commercially by the Great Northern Paper Co., the ranking newsprint producer in the United States.

6. *Semichemical Process.* The semichemical process, first developed by the Forest Products Laboratory, is suitable for pulping poplar, and possibly birch, for newsprint use without bleaching. Other hardwood species are bleachable at an additional cost. The process is being used increasingly for the production of printing papers other than newsprint. At the Richmond Pulp and Paper Co. mill at Bromptonville, Que., an average of 10% of the regular newsprint furnish consists of semichemical pulp.

7. *Cold Soda Process.* The cold soda process, which was also pioneered at the Forest Products Laboratory, has important potentialities for economic pulping of poplar and possibly other hardwoods for use in newsprint. While the process has not fully emerged from the developmental stage in this country, Cartiere Burgo in Italy, following completion of a successful pilot operation, is installing a small cold soda pulp mill to produce pulp for use in its regular newsprint production.

8. *Newsprint Trial Runs.* A series of pulping and paper-machine trials was conducted at the Forest Products Laboratory, Madison, Wis., in February, 1954. Cottonwood, a poplar species, was used as the basic hardwood raw material for these trials. Newsprint papers were made on the 13-in. paper machine of the Laboratory at a speed of 75 f.p.m. from furnishes containing cottonwood (and in some cases willow) groundwood blended with chemigroundwood, semichemical, or cold soda pulps made from cottonwood, together with semi-bleached kraft pulp in most cases.

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The feasibility of manufacturing newsprint at commercial speeds made from hardwood pulps in the proportions indicated should be conclusively determined by commercial trials on suitable equipment.

9. *Cost Considerations.* Aside from the broadened raw material base made possible by the use of hardwoods for the manufacture of newsprint, such use offers the prospect of important cost advantages.

10. *Further Research Needed.* More technical information is needed to establish the optimum proportions of hardwood pulps made by the various processes to obtain the best newsprint qualities at the lowest costs, and to determine the suitability of such pulping processes for a number of commercially available hardwood species about which relatively little is known.

*Hardwood Resources for Newsprint*

11. *Location of Hardwood Resources.* Most of the native hardwood timber stands in the United States are concentrated in the eastern part of the country. Of approximately 6.6 billion cubic feet of annual hardwood growth about 2.9 billion cubic feet is in the South, another 2.2 billion cubic feet is in the New England, Middle Atlantic, and Lake regions, and the balance in the Central and Plains States. Two important regions have been selected for special analysis in this report: (a) the hardwood bottomlands and other alluvial areas of the lower Mississippi Valley, and (b) the Northeast-Lake States region, covering New York, New England, Michigan, Minnesota, and Wisconsin.

*Lower Mississippi Valley*

12. *Hardwoods in the South.* The hardwoods in the timber stands of the South constitute a large potential supply of raw materials for pulp and paper manufacture. Of these hardwoods the cottonwood species offers a number of technological and economic advantages with respect to pulping for newsprint use.

The cottonwood tree, a poplar species, is considered the fastest growing commercial forest tree native to North America. It achieves a natural growth rate far in excess of that of pine, spruce, or other softwoods conventionally used in the manufacture of newsprint.

13. *Lands Suitable for Cottonwood.* Approximately 2.4 million acres of overflow and other alluvial lands in the region of the lower Mississippi Valley are estimated to provide favorable growing conditions and promising economic advantages for cultivation of cottonwood in commercial plantations. These lands are underutilized at present, particularly the unprotected or "batture" lands, where the danger of annual flooding makes agricultural cultivation impractical. Establishment of cottonwood plantations on these lands would greatly enhance their economic value to the owners and to the communities concerned.

14. *Growth Potential.* Such plantations give good promise of providing a substantially larger volume of useful wood at substantially lower unit costs than can be obtained in any other area of comparable size in the United States. There is evidence to indicate that on the acreage which provides favorable growing conditions for cottonwood, average annual wood yields of 400 cu. ft. per acre over a 15-year growing cycle, and 500 cu. ft. over a 25-year rotation, are attainable.

15. *Economic Feasibility.* The economic feasibility of commercial cottonwood plantations stems from (a) the marginal utilization of the batture lands, which are unsuitable for annual crops due to the danger of flooding, and (b) the rapid growth rate of cottonwood which produces an annual volume of wood far in excess of that produced in pine and spruce plantations. In addition to substantial quantities of pulpwood, cottonwood timber stands will yield very high values in veneer and saw logs when grown to mature dimensions.

16. *Mill Location.* The Memphis area and other locations on the Mississippi River are favorably situated with respect to cottonwood lands, provide economic access to major markets via barge transportation for a distance of several thousand miles on the most interconnected inland-waterway system in the country, and are suitable in other respects for the establishment of initial newsprint mills based primarily upon cottonwood.

17. *Pulp Potential.* If the full cottonwood growth potential on the estimated 2.4 million acres of suitable lands in the lower Mississippi Valley were realized, it could eventually provide raw material for at least 6.5 million tons of newsprint or other pulp production annually, assuming the use of only 30% of the annual timber growth for pulpwood. Realization of even a fraction of this potential in the form of newsprint expansion would significantly improve the domestic newsprint supply position of the United States, particularly in the South where newsprint consumption is growing more rapidly than in any other region of the country.

18. *Research and Field Trials.* Continuation of research should be encouraged, particularly with reference to determining the most profitable plantation management techniques under commercial conditions. Field trials are desirable to determine conclusively yields to be expected in commercial operation.

*Northeast-Lake States Region*

19. *Newsprint and Timber in the Region.* More than two thirds of the total United States demand for newsprint is in the Northeast and Lake States, which up to 25 years ago was also the center of newsprint production in the United States. Aside from Maine, manufacture of newsprint is no longer an important factor in the region, as growing scarcity of softwood timber and inability of producers to supply pulpwood needs at competitive costs have led to production shifts to higher grades of paper and migration of newsprint production to Canada.

Nearby sources of spruce, fir, and other softwood timber are seriously inadequate to meet the requirements of the pulp and paper industry in the region. Wood costs are high in comparison with other sections of the country, and steadily increasing. Significant expansion of newsprint or other pulp production in the Northeast-Lake States region is feasible only if based primarily upon use of hardwoods.

20. *Hardwood Stands.* Natural hardwood stands in the region, which are predominantly in farms and other small ownerships, now produce a substantial surplus of annual growth over drain in birch, beech, maple, and poplar species, which are suitable for newsprint use.

The volume of growth in these native hardwood stands could be significantly increased within a reasonable period if sufficient economic incentive existed for the management attention and effort required. Lack of such incentive in the past is primarily responsible for the generally understocked condition of these stands at the present time, which in turn has held annual growth far below production capabilities.

Availability of a broader market for hardwood pulpwood than at present, whether for the manufacture of newsprint or other uses, would help to provide the necessary incentive for improved hardwood management by making it economically more feasible to remove less desirable trees now hampering growth of the stands.

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21. *Pulp Potential from Surplus Growth.* The volume of hardwood timber resulting from present excess of growth over drain, plus increased volume of growth potentially available from application of general forest-management principles could, if fully utilized, provide an annual supply of birch, beech, maple, and poplar sufficient to produce about 8 million tons of newsprint or other pulps. The translation of a modest part of this potential into actual expansion of newsprint production would have a significant effect upon the newsprint supply-demand position of the region and of the United States generally.

22. *Poplar Plantation Possibilities.* Poplar plantations may be considered as a further potential source of hardwood suitable for newsprint use.

23. *Lands Available for Poplar Plantation.* Millions of acres of agricultural land in the Northeast-Lake States region, once under cultivation, are now unused or abandoned. About 2.5 million acres of land under scattered ownership, comprised largely of areas only recently withdrawn from agriculture, would be available and suitable for poplar cultivation. About one half of the acreage is in New York State where the unused agricultural land situation is most serious, but which at the same time offers the most promising opportunities for commercial forest plantation management.

24. *Pulp Potential from Poplar Plantations.* An average annual yield of approximately 375 cu. ft. per acre on a 25-year rotation would be potentially attainable on these lands. The potential supply of raw material from this source could ultimately support production of about 2.5 million tons of newsprint or other pulps annually.

25. *Future Commercial Development.* The commercial potentialities of poplar plantations as an economic source of timber have already been recognized by a number of large paper companies in the area. To broaden commercial interest, field trials are needed to confirm growth expectations and to determine the most efficient and profitable plantation methods to employ under commercial conditions.

Commercial fulfillment of the hardwood resource potentialities of the region will be materially influenced by the speed and vigor with which the practical business problems which exist are overcome.

Experience and ingenuity of the industry should provide the means of resolving these problems without serious delay.

* Presented by Jesse J. Friedman, Consultant, Department of Commerce, to the Committee on the Judiciary, House of Representatives, August 5, 1954.

## Ohio

### *The Straw Empires*

This is a story of finance, of facts and figures, of statistics motivated by cold cash. Not of papermakers and paper machines, not of the atmosphere in the old machine room with the roaming machine tender. Stories of the men and their famous old mills have been told; yarns have been spun and lives relived. These are integral parts, but this history cannot be complete without including an entirely different aspect of the development of the industry: The monopoly, the trust, and the men who "collected" paper mills; not in the least interested in papermaking but solely in the building of empires, "Straw Empires."

We call them "Straw Empires" for two reasons. First, because the empires existed for only relatively short periods of time. They were assembled from the remains of mills which, instead of being developed and modernized, were only exploited. Secondly, "Straw Empires" because straw-using board mills were involved almost exclusively.

This is the story of two such empires: The American Strawboard Co., and the Columbia Straw Paper Co. Mills in Dayton, Lima, Tiffin, Kenton, Piqua, Tippecanoe City, New Portage, and Hamilton were part of the first empire, the American Strawboard Co.; discussed in this first installment. It all started in 1889 when a number of old strawboard mills were brought together under the name of the American Strawboard Co. After several reorganizations these properties were taken over by the United Boxboard Co., and this firm, in turn, disposed of them in 1917. Here, it is virtually impossible to paint the entire picture of these organizations. Thumbnail sketches must suffice.

Clarke and Hawes, a partnership in Dayton, founded in 1853 was the earliest of a long string of board mills to be organized. The Lima Paper Mill in Allen County was one of the many mills incorporated in the trust. Constructed some time before 1879, it made five tons of strawboard and had a 62-in. one-cylinder machine. Benjamin C. Faurot, wealthiest man in Lima, was its president until 1890 when this mill became a part of the empire. This mill is famous for oil which was discovered there in 1885. A newspaper of that time reported: "Our city is still in a furor of excitement over the discovery of oil at the Lima Paper Mill. Not more than five feet of depth was gained yesterday, although the drills worked constantly. The oil is thick and brown in color. . . ." etc.

Kenton Paper Mills Co. in Kenton, Harding County, was established in 1882. An undistinguished mill with 15 tons of production, it became empire property in 1890 and was idle from 1897 on. The Piqua Straw Board and Paper Co. was on the other hand more interesting. Date of erection is unknown; the mill was part of the estate of one H. E. Loomis and was known as the hydraulic paper mill of Piqua. K. L. Wood operated the mill with his son until 1883 when the firm W. P. Orr and D. Spencer assumed control. As yet it has not been established whether there is a connection between W. P. Orr and today's Orr Felt and Blanket Co., also of Piqua. Six years later the empire took over. This mill did see some modernization. It was in its prime in 1899 when 30 tons were produced. The empire continued its hold until 1922 when the Piqua Straw Board Co., was formed, therefore becoming a sister mill of the old Tipp Paper Co. The new firm had offices in Akron and the Piqua mill continued to be operated until 1938 when, known as the Queen City Paper Co., it finally was reported idle.

The second mill just mentioned, Tipp Paper Co., was founded in 1883, taken over in 1890; it was run in the usual manner until 1902 when its 72-in. machine was enlarged from two to four cylinders. At the time of the death of the empire the Piqua Straw Board Co. took over. This mill's main name was also changed to Queen City Paper Co., and as such is known as the prosperous mill of today.

Another of "The Straw Empires" began with the Portage Straw Board Mill. O. C. Barber, magnate of the empire, collector of paper mills, apparently started with this mill in 1882. Barber was president in 1883; T. W. Cornell was vice-president. This was a somewhat larger mill, having two cylinders, one a 72-in., the second a 92-in. They made 24 tons, but did not manage to increase production till 1899. In 1902 they began to produce coated board but were closed definitely in 1910. This mill was first listed as being located in New Portage, Summit County. In 1900 it was "suddenly" located in Barberton! O. C. Barber was emperor.

> "The seasons change, the winds they shift and veer;
> The grass of yesteryear
> Is dead; the birds depart, the groves decay:
> Empires dissolve and peoples disappear."

Sir William Watson (1858–1935), author of the above, lived during exactly that time when industrial empires flourished. Perhaps he referred to precisely those types of empires which we are discussing.

Unable to bridge gracefully the span between the romance of poetry and the realism of papermaking for lack of space, we continue our tale of emperors and empires by telling of two additional mills which were part of the American Straw Board Co.

The American Pulp and Paper Co. of Tiffin in Seneca County was founded as a corporation instead of the usual "one-man show." Its earliest officers are obscure from the historian's point of view with the exception of its treasurer, Geo. D. Loomis, related to another Loomis of empire fame, whose mill in Piqua was taken over by the the American Straw Board Co. and operated until 1922.

This Tiffin mill was established in 1889, made 20 tons, and had a 90-in. one-cylinder machine. By 1891 it was one of the chain, and as such was apparently mismanaged into idleness seven years later. Started up again in 1902 it was operated, after slight beater room modifications, until 1910. The property was sold to a Pennsylvania organization in 1920, but was apparently never used again for its originally intended purpose.

Before turning to the second empire, brief mention of yet another "American Straw" possession, one still in existence and prospering: Today's Container Corporation of America, in Circleville. Believed to have had its origin in 1883, this mill was at one time the largest papermaking plant of its type in the country. The mill at that time had four cylinders and 22 beaters. One peculiar aspect of its operations was that it reportedly used loose straw, contrary to the practice of furnishing the rotaries with baled straw. One can just see the pitch-fork-swinging papermakers at that mill! Midwest Boxboard took over before 1920 and operated the mill till the thirties, when the Container Corp. assumed control.

The second major "Straw Empire," the Columbia Straw Paper Co., now warrants attention. One of the many mills controlled by this firm at one time or another was, what finally became the Xenia Paper Co. Constructed between 1879 and 1883 this was one of the few mills originally established by the empire itself. Unusual, too, was the fact that steam and not water was used from the start. Three tons of straw wrappers were produced here with a 62-in. cylinder. By 1907 production was gradually raised to an all-time high of 20 tons. In 1902, with the empire in woes and throes, Henry L. Beveridge, a former superintendent of the Beckett Paper Co. in Hamilton, took over the mill which carried his name for one year. In 1903 the name was changed to the Xenia Paper Co., with Beveridge, president. This man was also the founder of the Beveridge Paper Co. of Indianapolis, a well-known mill today. The Xenia mill met with misfortune in 1909 when it burned. It was never rebuilt. Another empire mill, the Defiance Paper Co., of the community by the same name, also ended up in a holocaust. Geo. D. Claflin, mentioned a number of times previously in this series, got into financial trouble with this mill. It went into the hands of a receiver and was later assimilated by Columbia. In 1899 fire destroyed this mill.

More flames of Newark, Licking County, where the Newark Paper Co., with a lilliputian 48-in. cylinder, had been established in 1881. Even this mill's 2-ton production was of interest to the Empire. In 1902 it characteristically burned.

There are others. Mills now practically unknown but at one time all empire property: the Massilon Paper Co., with two cylinders and a production of $1^1/_2$ tons, which rose to 15 tons and remained there until 1925; the Hastings Paper Co., in Springfield, Clarke County, which before 1883 enjoyed the unique name of Republic Printing Co., although always a conventional paper mill. Empire property for a short time this mill did not see the turn of the century. Judy, Knisely and Co., in New Philadelphia, Tuscarawas County, which changed names half a dozen times, indicating that not much could be done with it, eventually also ended up in the Columbia Straw Paper Co. fold.

Hinde and Dauch's mill in Sandusky also must list Columbia Empire in its family tree. Unfortunately space does not permit details.


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There remains what we believe to be the pioneer mill of Montgomery County, the Rutledge paper mill of Dayton, which was founded shortly before 1844 by Mark and George Rutledge (father and son). In 1864 the property was improved by construction of a new building. Further additions followed. By 1880 the property was two stories high and the mill used 6 tons of straw per day. Apparently real estate difficulties developed and the "royal" scavengers were able to secure a hold. Soon this historic mill, too, was listed as abandoned, more evidence that "Empires dissolve and peoples disappear."

—by Ernest F. Barker in *Ohio Tappi*.

### Water Control Developments

Growing public concern over the adequacy of current future water supplies is being evidenced in many regions of the country. Since future water stringencies will have considerable effect on paper industry and other waste treatment requirements a brief summary of recent developments in water control situations in various states is presented below.

*California.* Unsuccessful moves were made in the state legislature to obtain $75,000,000 to start immediate work on the vast $1,200,000,000 Feather River project. The whole project will await submission next year of a feasibility report.

In the meantime, a State Assemblyman announced plans to seek enactment next year of a $2^1/_2\%$ increase in the state sales tax to help finance water projects essential to the state's future development.

*Minnesota.* Possible amendments next year to the state public water law would tighten the law so that none of the state's 10,000 lakes could be pre-empted by persons buying up the land surrounding them and keeping the public off such lakes.

It is suggested that the state have the right to declare all waters in the state public, "under the police powers of the state for the welfare of the state."

*New Jersey.* New Jersey legislators will be asked to consider bond issue proposals for the erection of large dams and reservoirs in North and South Jersey which would add 400,000,000 gallons per day to the state's present safe yield of 415,000,000 gallons.

*New York.* The U. S. Supreme Court has given the City of New York the right to proceed with plans to take 300,000,000 gallons additional daily from tributaries of the Delaware River.

In the meantime, the New York City Citizen's Budget Committee has proposed that a study by outside engineers investigate the possibility of using waters of the upper Hudson rather than the Delaware on the grounds that prior claims of Philadelphia would make the Delaware water available only in "wet years" when it would not be needed by New York City.

*Ohio.* The newly formed Governor's Committee on Ohio Water Resources has begun studies to determine the adequacy of water supplies for industrial, agricultural, and municipal uses.

Reports from the state's division of water indicate that Ohio's underground water level is abnormally low with an accompanying adverse effect on stream flow.

*Oregon.* The State Water Resources Committee created in 1953 to study the state's water problem has gone on record in favor of a coordinated system of water resources development but has not yet reached a conclusion as to how such a program might be formulated and carried out.

*Pennsylvania.* Formation of a Pennsylvania State Water Authority to conserve available water resources and avert a threat to the growth of the Delaware Valley area has been proposed by the Greater Philadelphia-South Jersey Council.

The council's chairman declared that the water situation, particularly in southeastern Pennsylvania, "is crippling industrial and commercial potential and is posing a serious threat to our citizens."

*South Carolina.* Proposed legislation in South Carolina would put into effect a policy never before practiced in the Eastern United States.

This would replace the state's present riparian right doctrine. Under the proposed procedure no one could take water from a stream or watercourse without "valid right" and permission of a state board. To establish "valid right" use of the water for "useful industry or a well-recognized want" would have to be shown.

*Virginia.* Resolutions adopted by the state legislative council authorized the Virginia Advisory Legislative Council to continue its studies of water conservation problems and submit its findings and recommendations by Oct. 1, 1955.

*Mississippi.* A bill introduced in the State Legislature would study implementation of a water policy for municipal, industrial, agricultural, and all other beneficial purposes as a matter of public interest and welfare.

In supporting the measure, Representative Morrow quoted statistics on groundwater usages and stated that this source of supply is becoming more important with the advent of air conditioning and new industrial uses, such as wood pulp processing, the manufacture of chemicals, and other uses.

—from *General Bulletin* No. 47, National Council for Stream Improvement.

### *Dissolved Oxygen and Aquatic Life*

Supplementing the research conducted at The Institute of Paper Chemistry on the dissolved oxygen requirements of fish (reported in National Council for Stream Improvement *Technical Bulletin* No. 66), R. E. Dimick of Oregon State College has rendered a report on the same subject as it pertains to migratory fish common to the Northwest. Wild silver salmon exposed to oxygen tensions somewhat below 3 p.p.m. within the limits of 12 to 24°C. showed no mortality,

some surviving at as low as 1.4 p.p.m. Long-term experiments showed that the fish fed poorly and lost weight at an oxygen concentration of 2.0 p.p.m. but behaved normally and showed no ill effects after exposure to a 3 p.p.m. oxygen level 30 days. A technical bulletin covering these studies is being distributed to the Council membership.

### Pollution and Waste Treatment Notes

*Ohio*

At a meeting in Cincinnati, Ohio, held on June 22 a 13-man Industry Action Committee for the Pulp and Paper Industry was organized to cooperate with the Ohio River Valley Water Sanitation Commission. Virgil Minch of The Mead Corp. and E. W. Petrich of Howard Paper Mills were elected chairman and vice-chairman of the committee, respectively.

The first meeting of the committee will be in Cincinnati on Sept. 30, 1954.

As a means of cooperating with the Ohio Water Pollution Control Board, Ohio pulp and paper mills with the assistance of the National Council for Stream Improvement are currently developing a set of proposed standards for raw wastes for the Ohio pulp and paper industry.

Questionnaires showing water usage and waste losses have been received from member mills and these data have been tabulated and summarized by the National Council.

After the standards for each of the various broad groups of mills are developed and studied they will be submitted to the Ohio Water Pollution Control Board for approval.

*East Texas Pulp and Paper*

Facilities for treating the effluent from the new mill of the East Texas Pulp and Paper Co. are being constructed simultaneously with the mill near Evadale, Tex. These are designed to protect the water supply of the city of Beaumont and to prevent any serious load on the lower Neches River as a result of operation of this mill. Both structural and natural purification facilities are being employed in this system.

*Storage of Semichemical Liquor*

Attempts to keep spent neutral sulphite semichemical cooking liquor out of streams seasonally by storage lagooning have in some instances under adverse conditions resulted in odor problems arising from decomposition. It is believed that such decomposition would not occur at higher than normal liquor concentrations such as those which can be produced by screw-press washing and/or partial concentration. The Council's Virginia Polytechnic Institute project is determining if such is the case and if so, what the critical concentration is.

*Semichemical Liquor as a Road Binder*

Tests of considerable magnitude will be conducted this Summer by the Otsego Falls Paper Co. in Michigan to determine the effectiveness of neutral sulphite spent liquor as a road binder. Preliminary tests appeared promising.

*Kalamazoo Valley*

Treatment plants which have been placed in operation, or are in the process of construction by the paper industry in the Kalamazoo River Valley, were described on June 29 over WKZO-TV, Kalamazoo, Mich. Milton P. Adams, executive secretary of the Michigan Water Resources Commission, presented this information on a 15-minute television broadcast entitled "Michigan Unlimited" directed by Willis Dunbar. Pictures of the treatment plants were presented with commentary by Mr. Adams.

Four plants for primary treatment of deinking wastes, one chemical coagulation plant handling board mill wastes, and one installation for handling and storage of semichemical wastes for spraying on dirt roads are currently in operation.


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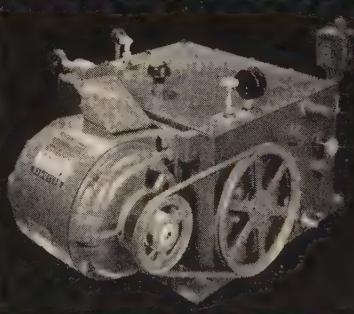

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Three additional deinking waste treatment plants, one plant handling rag mill waste, and two additional board mill treatment units (one flotation device, one settling tank) are in the process of construction.

The companies involved are as follows: St. Regis Paper Co., Rex Paper Co., Hawthorne Paper Co., National Gypsum Co., Mac-Sim Bar, Michigan Paper Co., Otsego Falls Paper Co., Sutherland Paper Co., Allied Paper Mills., Kalamazoo Paper Co. and Kalamazoo Vegetable Parchment Co. Facilities for treating sewage of the city of Kalamazoo are nearing completion.

*Southland Paper Mills*

Combined mill effluent purified by accelerated aeration and storage oxidation will be tested over an extended period at the Lufkin, Tex., mill to determine if its use for crop irrigation will affect the soil in any way. Two plots of 5.5 acres each, one being sandy and the other semitight bottom land, will be employed. Maximum fertilizer requirements as determined by soil analysis will be determined. Half of each plot will be irrigated with the maximum supplemented rainfall requirement from a sprinkler system. Records of crop yields and soil characteristics will be charted over a period of years.

*Indiana*

Enactment of legislation next year to strengthen Indiana's antipollution law has been advocated by State Conservation Director Doxie Moore.

*Roanoke River*

E. C. Hubbard, of the North Carolina State Stream Sanitation Committee, reported that a pollution study of the Roanoke River is now complete except for toxicity tests which it was hoped would be run this year. Mr. Hubbard reports that it will probably be next year, however, before the committee can hold public hearings to mix public opinion with scientific data as a prelude to classifying the river and directing polluters to lighten the load.

*Maine*

Classification surveys and hearings have continued during the past three months. Surveys have been completed on a considerable portion of the surface waters in Waldo, Knox, Lincoln, Kennebec, Hancock, and Washington Counties. Hearings have been held relative to surface waters entering tidewater in Hancock County and the Sheepscot and Medomak Rivers in Lincoln, Knox, Waldo, and Kennebec Counties. These hearings serve as a sounding board for local public opinion to assist the Water Improvement Commission in making its classification recommendations to the 1955 Legislature. Further hearings are anticipated in the near future on surface waters surveyed but on which hearings have not been held. Surveys are meanwhile progressing on surface waters in York, Oxford, Franklin, and Kennebec Counties.

It is anticipated that summarized and tabulated data gathered by surface water quality studies made throughout the state since late in 1950 by personnel of the Water Improvement Commission and of the Bureau of Health will be published this year as an addenda to the 1950 "Report on Water Pollution in the State of Maine."

—from *General Bulletin* No. 47, National Council for Stream Improvement.

## Paper for Packing Rubber

A recent issue of the *Natural Rubber News*, a monthly publication issued in behalf of the natural rubber growers of Malaya, notes that for certain uses where specialized packing is desired to avoid contamination of the rubber, paper packaging is being tried on an experimental basis.

A leading New York rubber dealer, recognizing that without some sort of cover certain types of rubber might be damaged and rejected by the purchaser, interested a large Amsterdam paper manufacturer in working out a suitable packaging for the rubber in question. As a result there have been several sizable shipments of this rubber received in paper bags.

The covers consisted of the following combinations: (a) polyethylene wrappings under multiwall kraft paper; (b) vinyl wrappings under multiwall kraft paper; (c) multiwall kraft paper coated on the inside to prevent adhesion.

The rubber bales themselves each weighed approximately 83–85 lb. and were about $^1/_2$ by 1 by 3 ft. in size. They received normal handling and were stowed both in the lower holds and in the 'tween-deck areas.

Very little distortion and no sticking of the coverings to the rubber was evidenced. In only two instances out of 216 bales had the packages been punctured and there was no visual evidence of dirt on the rubber in these two cases.

It is understood that several rubber manufacturers have shown an active interest in the tests and are willing to offer a slight premium for rubber packed in this manner.

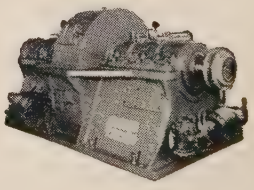
## Kraft Paper

Kraft paper is paper manufactured entirely from wood pulp made by the sulphate process, and in its most familiar form is the unbleached "brown wrapping paper" which comes in sheets or rolls and is used for packaging, for wrapping, and for making bags and other converted items.

Kraft paper is also available in various bleached and semibleached types which, when used in combination with dyes, give a variety of colors. Special finishes, stripings, and designs can also be made to order.

Kraft wrapping papers are papers which, according to grade, consist entirely, or in substantial part, of unbleached sulphate fiber. These papers, also according to grade, must meet certain specifications, including standard Mullen tests,

proven by performance



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and must possess certain qualifications as to composition, sizing, formation, etc. for their particular end use. The guaranteed grades must be clearly marked on the roll or bundle with a designation indicating that grade or guarantee.

Kraft wrapping papers are manufactured in two basic finishes; "Machine Finished" (M.F.) and "Machine Glazed" (M.G.). Machine finished kraft wrapping paper, which constitutes about 90% of the kraft wrapping paper used, is what is generally meant by "kraft wrapping." Machine glazed kraft wrapping paper has a shiny or glossy finish on one side, and usually has a striping within the paper, both of which give it a decorative appearance and make it more desirable for retail packaging in department stores, drug and candy stores, etc., whenever its lighter weights are not a detriment. Some companies can also furnish a "water finish" on order; this finish provides a shiny or glossy surface on both sides of the paper.

*Grades*

(*1*) *Standard Kraft Wrapping Paper* means any machine finished wrapping paper 25 lb. basis weight or over, containing 90% or more unbleached sulphate fiber and testing 70% or less of the basis weight Mullen test under standard TAPPI testing procedure.

(*2*) *No. 1 Kraft Wrapping Paper* means any machine finished wrapping paper 18 lb. basis weight, or over, containing 100% unbleached sulphate fiber and testing between 70 and 100% of the basis weight Mullen test under standard TAPPI testing procedure. All rolls and bundles of this grade must be designated with the guaranteed Mullen test or with the inclusion of the words "No. 1 Kraft."

(*3*) *Superstandard or Specialty Kraft Wrapping Paper* means any machine finished wrapping paper 18 lb. basis weight or over containing 100% unbleached sulphate fiber and testing 100% or more of the basis weight Mullen test under standard TAPPI testing procedure (point to the pound or better). All rolls and bundles must be designated with the guaranteed Mullen test, or with the inclusion of the words "Superstandard" or "Specialty."

(*4*) *Machine Glazed Kraft Wrapping Paper* means any kraft wrapping paper 18 lb. basis weight or over (usual range is between 18 and 40 lb. basis weight) glazed on one side by drying on a Yankee paper machine. There are no Mullen test requirements.

(*5*) *Standard Unbleached Kraft Butcher's Wrapping Paper* means any wrapping paper containing 90% or more unbleached sulphate fiber testing 70% or less of the basis weight Mullen test under standard TAPPI procedure, and possessing sizing formation, etc., to make it suitable for butchers' use. (See *Butchers' Paper*.)

(*6*) *No. 1 Unbleached Kraft Butchers' Wrapping Paper* means any wrapping paper containing 100% unbleached sulphate fiber, testing 70% or over of the basis weight Mullen test under standard TAPPI testing procedure and possessing sizing, formation, etc. to make it suitable for butchers' use. All rolls and bundles must be designated with the guaranteed Mullen tests or the words "No. 1 Kraft Butchers."

*Miscellaneous Information*

(*1*) *Basis Weight.* The weight in pounds of 500 sheets, 24 × 36 in., or 864 sq. in. each.

(*2*) *Rolls.* (a) Counter rolls are 9 in. in outside diameter and standard widths in M.F. finish of 9, 12, 15, 18, 20, 24, 27, 30, 36, 40, 48, 54, 60, and 72 in. Counter rolls come in the following standard widths in M.G. finish: 6, 9, 12, 15, 18, 20, 24, 27, 30, 36, 40, and 48 in. (b) Standard (or industrial) rolls are 12 to 18 in. in outside diameter and come in standard widths of 30, 36, 40, 48, 54, 60, and 72 in. (c) Jumbo rolls (or mill reels) are 26 in. or more in outside diameter and are too large for use with a wrapping paper stand. Jumbo rolls are usually purchased for rewinding, sheeting, or other converting operations. All manufacturers do not make all widths.

(*3*) *Sheets.* Standard size sheets are: 10 × 15, 12 × 18, 15 × 20, 18 × 24, 20 × 30, 24 × 30, 24 × 36, 30 × 40, 40 × 48, 48 × 60, 48 × 64, and 60 × 80 in. All manufacturers do not make all sizes.

(*4*) *Bundles.* Sheets are usually packed, either flat or soft fold (interleaved without crease) in bundles weighing 100 lb. in sizes smaller than 40 × 48, and in 150-lb. bundles size 40 × 48 and larger. The smaller sizes are usually packed flat, the medium sizes flat and soft fold, and the larger sizes soft fold.

(*5*) *Special Weights and Sizes.* Orders for special weights and sizes are usually required to fit machine trims in the same grade, basis weight, and diameter of rolls, and usually require a minimum quantity of 10 tons or more.

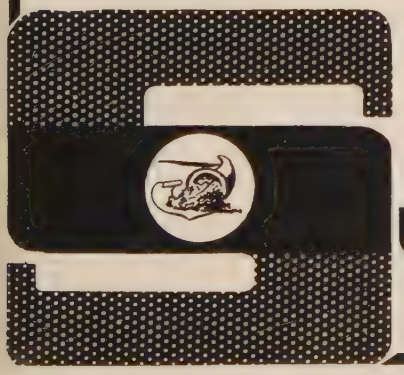
(*6*) *Billing Weights.* All kraft wrapping paper is billed at the scale gross weight including wrappers, twine, and plugs.

(*7*) *Commercial Delivery.* All shipments of kraft wrapping paper are considered good commercial delivery when the actual basis weight does not exceed 5% above or below the specified basis weight.

All shipments of kraft wrapping paper in excess of 5 tons are considered good commercial delivery if the over delivery or under delivery does not exceed five per cent of the specified tonnage. All shipments of 5 tons or less are considered good commercial delivery if the over or under delivery does not exceed 10% of the specified tonnage.

(*8*) *Formula for Computing Weight and Yardage.* (a) To determine approximate weight or number of pounds in a roll of kraft wrapping paper multiply the width of the roll by the factor for a roll of the same diameter.

Example: the factor for 9-in. diameter rolls in all grades is 1.5. Therefore, the weight of a 10-in. wide roll, 9 in. in diameter would be 10 × 1.5, or 15 lb.

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performance

Sutherland High Yield Kraft System*

SUTHERLAND REFINER
CORPORATION

* U.S. Patent No. 2,591,106

TRENTON 8, NEW JERSEY

Factors for several of the more common diameters are listed below:

| | |
|---|---|
| 9 in. diameter, 1.5 factor | 24 in. diameter, 10.7 factor |
| 10 in. diameter, 1.8 factor | 26 in. diameter, 12.6 factor |
| 12 in. diameter, 2.7 factor | 28 in. diameter, 14.6 factor |
| 15 in. diameter, 4.2 factor | 30 in. diameter, 16.7 factor |
| 18 in. diameter, 6.0 factor | 34 in. diameter, 21.5 factor |
| 20 in. diameter, 7.5 factor | 36 in. diameter, 24.0 factor |
| 40 in. diameter, 29.7 factor | |

(b) To determine number of lineal yards in a roll of kraft wrapping paper, use formula given below:

Weight of roll × 12,000 ÷ width of roll × basis wt. = approximate lineal yards per roll

(*9*) *Marking and Labeling.* Packages are not marked, stenciled, or labeled by the manufacturer with other than the actual dimensions, basis weight, grade, form, and trade name of the paper contained.

—from *American Paper Merchant*, July, 1954.

### Mummy Paper

Apparently little is known of the experiments of I. Augustus Stanwood (born Dec. 7, 1839) in the manufacture of paper by the groundwood process. Stanwood and William Tower began their papermaking in Maine in 1863 and it is claimed by the Stanwood family that their mill produced groundwood paper in January of that year.

The most interesting phase of Stanwood's career, however, was his use of Egyptian mummies for making wrapping paper. The information here set down regarding the mummy paper was given to me by Stanwood's son, Daniel, a retired professor of international law, living in Massachusetts. During the Civil War, according to Professor Stanwood, his father was pressed for raw material to keep the Maine mill in operation and he had to use his ingenuity to overcome the difficulty. This he did by importing mummies from Egypt for the sole purpose of stripping the dried bodies of their cloth wrappings and using the material for making paper. Professor Stanwood informed me that his father brought several shiploads of mummies to his mill in Gardiner, Me., and threw the woven wrappings as well as the papyrus filling into beaters and manufactured from these substances a coarse brown wrapping paper, which eventually found its way into the shops of grocers, butchers, and other merchants who used paper of this kind. It was further stated that the rags stripped from the long-dead Egyptians caused an epidemic of cholera among the rag-pickers and cutters in the Maine mill, for at that period there was no regulation regarding the disinfection of rags. Professor Stanwood also related that the only competition his father encountered in purchasing the mummies was the Egyptian railroad, for during a ten-year period the locomotives of Egypt made use of no other fuel than that furnished by the well-wrapped, compact mummies, the supply of which was thought at the time to be almost unlimited. Stanwood the papermaker died March 6, 1914, and in the various obituaries published by New York newspapers, accounts were given of his experiments with groundwood and his unusual manufacture of wrapping paper from the wrappings of Egyptian mummies.

—from "Papermaking," by Dard Hunter.

### Mullen Tester

The Mullen tester is the oldest piece of standard equipment used for paper testing. It is used for determining the bursting strength of materials in sheet form. The Mullen tester is still manufactured by B. F. Perkins & Son, Inc., of Holyoke, first manufacturer of Mullen's invention.

Inventor of the Mullen tester was John William Mullen born in 1849 in West Fitchburg, Mass. In manufacturing all his life, Mullen devoted most of it to papermaking. He superintended the construction of a paper mill at Augusta, Me., and ran a paper company at East Pepperell. After moving to Michigan, he formed the Mullen Brothers Paper Co. at St. Joseph, Mich.

### Water and Sewerage Plant Papers

Louisiana State University and Agricultural and Mechanical College, Baton Rouge, La., has published *Engineering Experiment Station Bulletin* No. 44, Proceedings of the Seventeenth Annual Short Course for Water and Sewerage Plant Superintendents and Operators, 1954.

### Air-Drop Research

The Military plans to streamline its method of aerial delivery of supplies in the interest of efficiency and economy. To reduce costs and excessive losses, the Quartermaster Food and Container Institute has entered into a research contract with the University of Texas, Austin, Tex., to develop and test improved shock-absorbing devices which can be used in the air drop of cargoes of various weights.

### Petroleum Packaging

Packaging Institute, 342 Madison Ave., New York, N. Y., has issued a report on the Standard Specifications for Containers and Pallets recommended by the Petroleum Packaging Committee. The report is available at $1.00 per copy.

### Western Michigan

Robert N. Zabe, formerly a member of the research staff of Champion Paper and Fibre Co., Hamilton, Ohio, has been named associate professor in paper technology at Western Michigan College.

### Syracuse

Charles Luner and Robert L. Gilbertson have been appointed to carry on special studies at the State University

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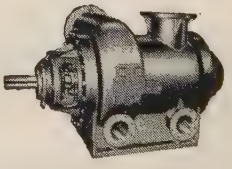
Sutherland Breaker Trap

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of New York College of Forestry, Syracuse, N. Y. Mr. Luner is assisting Michael Sware of the college in his studies on the fundamentals of polymers. Mr. Gilbertson is working on the anatomical phases of the chemical debarking studies being carried on at the college.

Robert Zabel will continue his studies of paper mill slimes under a renewal of a $2500 grant to the college from the American Mining and Smelting Co.

American Viscose Co. of Philadelphia has awarded two $2000 fellowships to the college. Recipients are Edgar A. McGinnes who will study wood technology, and Robert Lenz, who will work on cellulose chemistry problems.

### Lowell Tech

Donald F. Bullock of Ware, Mass., is this years' winner of the McLaurin-Jones Scholarship, awarded by the McLaurin-Jones Co., Brookfield, Mass., at Lowell Technological Institute, Lowell, Mass.

### Timber Harvest

Timber harvested from national forests during the year ended June 30, was an all-time record 5,370,022,000 bd. ft., according to August 4 release of U. S. Dept. of Agriculture. This represents an increase of 4.07% over last year's record of 5,160,355,000 bd. ft. Greatest increases in timber cut were reported from forests in Montana and Idaho where access roads have been built to salvage insect infested timber. Forest Service region one, made up of Montana and northern Idaho, increased timber cut from 574,255,000 bd. ft. in 1953, to 648,091,000 bd. ft. in the year ending June 30. Value of timber taken from the national forests showed a drop of about $1.50 a thousand bd. ft., reflecting lower prices throughout the country. Total value of timber harvested, therefore, dropped from $70,616,025 in 1953 to $65,407,400 in 1954.

### Southern Institute of Forest Genetics

Berch W. Henry has been appointed head of the new Southern Institute of Forest Genetics which the Southern Forest Experiment Station is establishing in southern Mississippi. The Institute is to serve the entire South, from Virginia to Texas. It will investigate fundamental problems in forest genetics, do applied research in tree selection and breeding, and serve as a demonstration area and source of information for forest genetics research and application. The Institute will in no way compete with other genetics work already being done by existing private, State, or federal agencies Instead, it is intended to supplement and assist such work.

The Institute's headquarters will be at the Harrison Experimental Forest, about 20 miles north of Gulfport, Miss. on Highway 55. The Harrison Forest was chosen from many other locations in the entire South as being the best place for selecting or developing improved strains of the four major species of southern pine. Offices, a laboratory, and a greenhouse were built on the Forest during CCC days and will be expanded to serve the Institute.

F.P.L.

By breeding and growing top-quality pine trees much as we now breed top-quality beef cattle, they can be tailor-made for many purposes. Some pulpwood pines, for example, can be improved to yield double the amount of kraft pulp per acre per year.

Forest Products Laboratory (Madison, Wis.) Report No. 1993, "Greater Pulp Yields Per Acre Per Year," explains this theory, which is based on the premise that the density of wood is the best single index to wood quality. By controlling density you improve most of wood's properties for lumber. Greater density increases the yield of pulp possible per cord of wood.

Two other reports just issued by the Laboratory deal with fundamental aspects of tree growing for wood quality. They are: "Controlling the Proportion of Summerwood in Longleaf Pine," Report No. 1988; and, "Specific Gravity Relative to Characteristics of Annual Rings in Loblolly Pine," Report No. 1989.

Four new reports in the Laboratory's series of Information Leaflets on foreign woods have also been issued. Data are given on the mechanical properties of the wood, seasoning and shrinkage characteristics, durability, and working qualities.

Report No. 1960 describes manbarklak, a wood native to Brazil, Trinidad, and Costa Rica. This wood is considered good for marine construction in sea water because of its durability. It is a hard, dense, strong wood with mechanical properties that exceed those of white oak.

Report No. 1985 describes conacaste or guanacaste, another tropical American wood that is hard and heavy but is only fairly durable. The wood is easy to work, finishes smoothly, and is considered a possible substitute for yellow-poplar in plywood core stock.

Report No. 1987 describes klinki, a softwood grown in Papua and New Guinea and sold in the United States. Klinki compares favorably with ponderosa pine. It is easy to work, takes a smooth finish, and has excellent dimensional stability because of its low shrinkage.

Report No. 1991 describes carapa, a wood found in the West Indies and in Central America. Carapa is easy to work on most machine tools, and it peels well for veneer.

Paint difficulties caused by rain that gets behind siding can be reduced by use of water-repellent preservatives to keep the water out, according to Report No. 1990, just issued by the Laboratory. The water-repellent preservative can be applied by brush or the wood siding can be dipped in the chemical.

The following is the complete list of new reports issued by the Laboratory:

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performance

Sutherland Inc. Pressure Washing*
Sold and Serviced by

PRESSURE · WASHING ·

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*U. S. Patent No. 2,355,091

"List of Publications on Mechanical Properties and Structural Uses of Wood and Wood Products," Report No. R200.
"Color Tests for Differentiating Heartwood and Sapwood of Certain Oaks, Pines, and Douglas-fir," Technical Note No. 253.
"List of Publications on Wood Finishing Subjects," Report No. 454.
"Some Machining Properties of Engelmann Spruce," Report No. 1944-7.
"Manbarklak," Report No. 1960.
"Hollow-Core Flush Doors," Report No. 1983.
"Polyhydric Alcohols from Wood," Report No. 1984.
"Conacaste or Guanacaste," Report No. 1985.
"Klinki, Rassu, Pai," Report No. 1987.
"Controlling the Proportion of Summerwood in Longleaf Pine," Report No. 1988.
"Specific Gravity Relative to Characteristics of Annual Rings in Loblolly Pine," Report No. 1989.
"Water-Repellent Preservatives Reduce Rain-Caused Paint Blistering on Wood Siding," Report No. 1990.
"Andiroba, Crabwood, Cedro Macho, Carapa," Report No. 1991.
"Glued and Nailed Roof Trusses for House Construction," Report No. 1992.
"Greater Pulp Yields Per Acre Per Year," Report No. 1993.

### Paper Pallets

Present methods of palletizing both bagged and packaged commodities are challenged by American Cyanamid's development of the Accopak Pallet. The new paper pallet, already in use for palletizing company's shipments of phthalic anhydride, offers important time and money saving advantages in shipping and in storing.

The Accopak Pallet features simplicity; it consists of two durable cardboard tubes and a strong paper sling made of Melostrength resin-treated paper, designed to impart unusual wet and dry strength. Two bayonet type forks, also designed by American Cyanamid and which attach to any fork lift truck, slip into the tubes. This enables the palletized load to be lifted, carried, and stacked in single, double, or triple-deck tiers without removal of the pallet during storage or shipment. One pallet is capable of holding 24 80-lb. bags.

An important advantage which this method of palletizing offers is in facilitating the loading and unloading of freight cars, says American Cyanamid. The sling is no thicker than heavy wrapping paper. The loading of a shipment palletized the Accopak way saves between 8 and 10 man-hours as compared with a nonunitized method. The palletized traveling also helps bags to retain a more compact shape. Thus they stack more neatly and occupy less space.

The wooden pallets usually required for warehouse storage can also be eliminated by use of Accopak Pallet. As in the railroad car, the bags can be stacked in multiple-deck fashion, pallet remaining with its unit.

Pallets themselves present no storage or assembly problem. They can be rolled up like maps or stored flat; accompanying tubes form a convenient size bundle.

Development of the Accopak Pallet is a by-product of American Cyanamid's study of materials handling techniques and also its research in field of paper chemicals. The company will patent the new pallet, and manufacturing licenses will be offered to companies in the paper industry.

### Pentagon Pulper

Downington Manufacturing Co., Downington, Pa., has contracted to build a pulper and wet machine for the Pentagon in Washington, D. C. The installation will be used to destroy secret Government documents by converting them into sheet pulp when they have served their purpose. At present the papers are burned. The new disposal method will not only assure complete disintegration of confidential security papers, but will also produce a readily salable by-product in the form of pulp. In addition, the installation will remove fire hazards and other difficulties that had resulted from burning the papers.

### Gair

A modern new boiler house has been erected at Rober Gair Co.'s Haverhill, Mass. plant, designed by its engineerin department in New York City. This new steam and powe installation, which cost $1,200,000, makes the plant wholl self-supplying. The equivalent of an 8-story building, it i 83 ft. long, 60 ft. wide, and 90 ft. high above the basemer floor. This building has been designed to house the boile feedwater pumps and the chemical treating equipment for th boiler feedwater. The huge water softener and deaerato 44 ft. tall, is capable of handling 200,000 lb. of water an hou in the deaerator and 75,000 lb. per hour in the water softene Make-up water is pumped from the adjacent Merrimac River.

The building has a heavy structural steel frame, walls c varicolored face brick, with reinforced concrete floors an roof deck. An elevator has been installed to facilitate mair tenance and handling of materials.

Installed in the building is the new boiler 60 ft. high capable of delivering 100,000 lb. of steam per hour of super heated steam at 610 p.s.i. and 650°F. Steam from this uni is delivered to a new 2500-kw. topper turbine, located in th existing engine room, which exhausts into the steam mai formerly supplied by the old boilers at a pressure of 165 lb The boiler is coal fired by two pulverizer units, each of whic has a capacity of 6700 lb. of coal per hour.

One novel feature is that the coal is not stored in the base ment but at the top of the building. Coal drops from railroa cars into a coal hopper under the track. A belt conveyo carries it into the building and dumps it onto a bucket ele vator, adjacent to the passenger elevator. Coal is hoisted t the top of the building, then discharged onto another bel conveyor which carries it to a coal bunker, 30 ft. deep an about 50 ft. long, which holds 425 tons.

Coal is drawn off the bottom of the bunker through an one of five gates, onto a third belt conveyor leading to auto matic scales. The weighed load drops through chutes to controlled pulverizer system, from which the coal is blow into the burners. The whole operation is mechanical. As from the furnace is conveyed to a large silo from which it ca be unloaded into trucks for disposal.

The modern design and greater efficiency of this new in stallation produce steam and electricity at lower cost. Th power plant, with its new boiler, turbo-generator, and auxi iary equipment, reduces operating expense while at the sam time increasing steam and electrical capacity. It also elim nates the expense of purchasing some of the power on the out side as was previously done.

Homer Murphy has been appointed sales manager for th Angelus Paper Box Div. of Robert Gair Co. at Los Angeles Calif.

### Cellu-Products

Cellulose Products Co., High Point, N. C., will open a new plant at Lenoir, N. C. The company produces "Cellucrepe," and at present operates plants at High Point and Martins ville, Va.

### St. Lawrence Corp.

St. Lawrence Corp.'s new kraft machine at Red Rock, Ont is reported to be producing at well over anticipated schedul

P. M. Fox, president, in his address to company share holders revealed that since the new machine came into pro duction March 15, output has been in the neighborhood of 7 tons higher per day than allowed for in the original productio schedule. May production, estimated at 250 tons, wa actually running at 325 tons per day. Capacity productio will be reached in September.

Indications are, said Mr. Fox, that St. Lawrence will be ab to ship its total kraft tonnage from the Red Rock and Ea Angus mills during 1954.

### Building Products

The first paper mill of its kind between Red River, Manitoba, and the Pacific Coast, Building Products' new $1\frac{1}{2}$ million plant has gone into operation in Edmonton, Alberta.

With the new manufacturing unit producing all the dry felts and dry sheathings required by the company's adjacent roofing mill for the manufacture of asphalt shingles, roll roofings, and allied products, Building Products now has a completely integrated operation.

In addition, the paper mill will produce other types of paper products for industrial use throughout Alberta, replacing similar products previously brought in at heavy transportation costs from distant points. Company officials said the mill will consume a certain amount of local pulpwood and will provide an outlet for secondary materials such as rags and wastepapers where formerly no regular outlet existed.

The plant, initially geared for production of 20,000 tons of paper products per annum, is capable of considerable expansion as the city, province, and the West develop.

The papermaking machines, which will produce paper with a trim of 115 in., are of multi-cylinder design, allowing for the necessary flexible operations for producing a wide variety of paper products. The latest equipment available for the preparation and handling of various materials used in papermaking will be used.

### Powell River

Achieving a record-breaking speed of 2001 f.p.m., Powell River Co.'s No. 8 machine attained in July the goal for which papermakers have been striving during the past decade.

This speed was maintained for over $\frac{1}{2}$ hr., during which time Powell River technicians and operating staffs studied the new problems created. Many valuable facts were discovered which will enable this and possibly an even greater speed to be maintained in the future.

Prior to crashing the "miracle" 2000-ft. goal, the machine had operated at 1950 ft., which also surpassed any previous paper machine speeds. No. 8 machine of Powell River was the first to install and maintain couch transfer equipment.

The 2000-ft. record is to the papermaker what the breaking of the 4-minute mile and the cracking of the sound barrier were to sport and science. The late George F. Steel of New York once recalled a "gigantic" paper machine of the 1880's which produced 10 tons a day at speeds of 100 f.p.m. Up until 1930 no paper machine had reached the 1000-ft. mark.

The original Powell River paper machines operated at 660 f.p.m. from 1912 right through until 1930. In the next 10 years speeds up to 1500 f.p.m. were recorded; and in the last decade pulp and paper producers have been steadily speeding up machines beyond this limit.

Powell River's No. 8 machine, designed for 2000-ft. speeds, has crept up to 1700 ft., and in recent months passed the 1800-ft. mark. It calls to mind "speed records" of the early days and the long struggle before present speeds were attained.

Powell River papermakers say they are confident that with necessary alterations, and the operating knowledge they now possess, speeds in excess of the 2000-ft. mark are possible.

### Champion-International

The first beta-ray reflection gage to be installed on a wide papermaking machine in the East is now in operation at Champion-International Co., Lawrence, Mass.

This ultrasensitive noncontact measuring instrument has an element of thallium as its radioactive isotope and is installed on a 166-in. papermaking machine. The gage scans the width of the web at intervals of approximately 4 min. giving a continuous measurement, both lineally and across the width, of the basis weight of the paper being delivered.

The gage is a product of Industrial Nucleonics Corp. of Columbus, Ohio, and approval for its installation was granted by the Atomic Energy Commission.

The new beta gage, known as an AccuRay, is of the reflection type, which is designed to give accurate thickness readings of the web of paper. The working principle of the gage is based on the fact that a certain percentage of high-speed electrons emitted by the radioactive isotope above the paper will reflect back into the detector unit. These reflected radiations are then transmitted electronically to a recording device where they are transcribed on a scale chart. The mechanism is set for the particular thickness or weight of paper to be made and any variations in this thickness will be immediately recorded on the chart. The immediate detection of the variations permits prompt adjustment of the paper machine, thereby guaranteeing uniformity of the paper being manufactured.

### Charmin

Charmin Paper Mills, Green Bay, Wis., has established a cooperative program with the U. S. Forest Service to provide scholarships for 34 Green Bay boys to attend the Trees for Tomorrow camp at Eagle River. This is the fifth consecutive year of the program with Charmin paying all expenses for the three-day session at the camp. Biology instructors in Green Bay's three high schools include this annual program in their curriculum. The Forest Service runs the camp, and instruction is given in conservation in forestry and wildlife.

### Scott

*Everett*

Continuing expansion of Scott Paper Co.'s West Coast Div. on Puget Sound at Everett, Wash., was highlighted recently by the start-up of No. 2 paper machine. The first papermaking machine went on the production line in December of last year.

In conjunction with the additional production that will come from the second high-speed machine, finishing equipment has been installed to manufacture several more Scott products. The advent of additional brands at Everett brings closer to realization the manufacture of the entire family of Scott products on the West Coast.

Other major areas of activity include construction progress on the fourth floor of the converting building and work on the new engineering, maintenance, and cafeteria building.

***Scott Paper Co.'s West Coast Div. plant, located on Puget Sound, Everett, Wash., is pictured above. Left, background, is the pulp mill, world's largest bleached sulphite operation, from which pulp is piped in slush form to new paper mill, right foreground. Present construction of papermaking facilities includes section of building at left extending toward water which houses two new paper machines. Three floors of new converting building in foreground also are in use for finishing of Scott products. Two additional paper machines will be installed in new building to be constructed in area behind converting building and next to existing paper mill***

able to other landowners and tree growers, and disseminated through technical publications. For the ultimate goal of the Research Dept. is to increase the yield of southern pine from southern land, and thereby add to the region's growth, economy, and standard of living.

Union Bag is now making paper out of wood chips, bought direct from a lumber company, that were formerly considered normal waste in a sawmill operation.

Instead of allowing its slabs and edgings to be burned as waste, the F. W. Darby Lumber Co. of Statesboro is chipping them up and sending them to Union Bag's Savannah mill by the box car load.

The first paper was made out of these "ready-bought" chips in July of this year.

For the past several months, the Woodlands Div. and the Industrial Engineering Dept. have been working with the Darby Lumber Co. in ironing out the details for the waste utilization project. The industrial engineering was done under the direction of C. D. Blake with Art Bunker as project engineer.

Equipment installed at the lumber mill includes a mechanical log debarker, a chipper, a chip screen, and conveyors for handling both logs and chips. In producing the chips, the first step is to remove the bark from the logs in the mechanical debarker. The bark-free logs are then sawed up into lumber, and the slabs and sidings removed in this trimming process are fed into a mechanical chipper.

From the chipper the chips are carried directly to a box car by conveyors, and, after transit, unloaded directly at the Union Bag mill.

Union Bag is presently negotiating for the purchase of chips from waste material from several other large sawmill operations in this section.

### National Gypsum

The National Gypsum Co., Buffalo, N. Y. has promoted Leonard L. Hank to the newly created position of general production manager in charge of gypsum, paper, and insulation board plants. Other promotions on the company's production staff include Eugene W. Odenwaldt to the new position of general production manager in charge of paint, rock wool, lime, asbestos, and metal lath plants; and Charles H. Dwyer to director of plant industrial relations.

### Flambeau

Sulphite roadbinder promises to provide high quality runway surfaces at costs that small airports can afford. The Park Falls, Wis., airport treats its runway with roadbinder obtained from the Flambeau Paper Co. which uses this pulp mill by-product for the dual purpose of road improvement and keeping it out of the stream.

Original purpose of the first roadbinder application just after the airstrip was regarded last Fall was to get rid of dust which annoyed the neighbors and caused extra maintenance on engine intakes. Applied at a rate slightly less than 2 gal. per square yard, the roadbinder not only laid the dust, but also bound together the gravel and finer materials into a hard surface comparable for traction and smoothness with blacktop or concrete. The better rolling surface permits faster take-offs and bigger payloads from the 1800-ft. runway than would be possible on either grass or plain gravel, and has brought many compliments from local and visiting pilots.

### Abitibi

The Hon. C. Mapledoram, Minister of Lands and Forests for the Province of Ontario, recently inaugurated a 40,000-acre Woodlands Laboratory established by Abitibi Power & Paper Co. Ltd., 80 miles north of Port Arthur, Ont., for research and experiments designed to increase per-acre pulpwood yield.

Abitibi established the laboratory to experiment with modification of logging methods and other treatments de-

*C. B. Davis, vice-president (Woodlands) Abitibi Power and Paper Co., Ltd., (right) presents the Hon. C. Mapledoram, Ontario Minister of Lands and Forests, with a bronze plaque as a souvenir of the inauguration by the Minister of a 40,000-acre Woodlands Laboratory located 80 miles north of Port Arthur*

signed to improve quantity and quality of regeneration and, secondly, to study other ways of securing the maximum yield of which the forest is capable. The objective of the laboratory is to find out what methods can be applied to the solution of these problems within practical and economic limitations. It is a pilot plant or an applied research center, rather than a project of basic or fundamental research, although facilities can be made available for other agencies to carry on such work.

The laboratory is located on G.T.P. Block III, about 60 miles northwest of the Lakehead. It is crossed by the Trans-Canada Highway, the C.N.R. and the C.P.R. lines. The location was chosen because it was company freehold land and because rail delivery, rather than water, permits utiliza-

*Scotch pines were planted by official guests at the inauguration of the 40,000-acre Woodlands Laboratory, established by Abitibi Power & Paper Co., Ltd., 80 miles north of Port Arthur, Ont. Left to right: H. H. B. Styffe, president, Northwestern Ontario Timber Operators' Association; J. G. Somers, Provincial Forester, Province of Manitoba; the Hon. C. Mapledoram, Minister of Lands and Forests for the Province of Ontario, who formally inaugurated the laboratory; C. R. Mills, manager, Ontario Forest Industries Association; J. W. B. Sisam, Dean, Faculty of Forestry, University of Toronto; F. Smith, president, Fort William Ontario Chamber of Commerce; Mayor E. G. Carson of Fort William; P. Neal, acting Mayor of Port Arthur, Ont.; Murray Fleming, C. D. Howe Co., Fort William, Ont.; C. B. Davis, vice-president (Woodlands), Abitibi Power & Paper Co., Ltd.*

tion of all species that may be encountered. The company could scarcely hope to obtain a single, fairly small area which represented every forest condition on its limits, but it wanted and managed to get an area which carries "problem forest" over most of its area. Other important factors in selection were to obtain an area with good natural protection features, and an area with a fair proportion of immature forest, for it is here that silvicultural measures have the best chance of being successful and of paying their way.

The area set aside for the laboratory is roughly 40,000 acres, comprising the westerly two thirds of G.T.B. Block III. This block is one of ten originally granted to the Grand Trunk railway in 1905 by the Province of Ontario as a construction subsidy. Abitibi leased cutting rights from the railway from 1925 until the blocks were sold in the last few years to the company.

Of the total area the first unit of 20,000 acres has been developed. For this purpose eight miles of all-weather road, a ten-car double-end loading spur, and a portable camp to house 25 men have been built. The organization is set up to operate on a year-round basis and a permanent crew will carry on all phases of the work to be done.

An advisory council made up of the head office and divisional foresters of the company meets once or twice a year in the forest to discuss and plan details of the work under way or proposed. While the laboratory is a head office project, it is assisted materially by the Lakehead Div. in the matter of purchasing, freighting, scaling, and accounting.

The scope of the project has been set for the present at 3000 cunits annually as this is considered in keeping with the sustained yield possibility of the first unit. This rate of production has been maintained since the project started in August, 1952.

The equipment allocated to the project includes a five-ton general purpose International Truck, a D-6 tractor with bulldozer and winch, a station wagon, horses, sleighs, and the regular camp, logging, and fire protection equipment and light plant, etc.

The forest in the first unit includes about 5000 acres of 70 to 80-year-old stock, with stands of pure poplar, spruce, and jackpine and mixtures of these species with birch. Another 1500 acres is unclassified cut-over and the remainder is 40-year-old stands of jackpine with spruce and muskeg. Some 30,000 jackpine ties were cut in 1927, and since that time some 30,000 cunits of pulpwood had been removed up until the laboratory was established.

The major problems being attacked are the encouragement of advance growth and regeneration, and improvement of species composition. The methods being tried include release cuttings, thinnings, improvement cuttings, partial cuttings by individual trees, strips, groups and wedges, underplanting and direct seeding, where the need for these is indicated.

For purpose of planning and record the area is broken into compartments of about 400 acres, and these are broken down into subcompartments of from 1 to 50 acres, depending on condition and treatment required.

The preliminary plan was to take one third of the volume of each compartment in each of three cuts spaced at 5-year intervals. This may, of course, require revision as results of treatments become apparent.

### Mando

The world's two largest emblems made of "Scotchlite" brand reflective sheeting have been erected on a 150-ft. water tower near the Canadian border by the Minnesota and Ontario Paper Co., International Falls, Minn. Each emblem is 25 ft. wide and 22 ft. high.

The reflective sheeting was used for economy's sake, M and O officials said. With reflective sheeting the initial installation was several hundred dollars less expensive; and the long life of the material is expected to result in saving several hundred dollars more—emblems made of other materials would require more frequent replacement and maintenance.

The emblems were adhered to the water tower by the weatherproof pressure-sensitive adhesive on the back of the reflective sheeting. The smooth surface plastic material partially cleans itself in rain. The emblems were made in the M and O company's art department using special opaque black and transparent green colors. A transparent plastic coating was brushed over the completed emblems to add to the luster and durability.

### Chips from Sawmill Slabs

Wholesale recovery of pulpwood chips from both large and small mill slabs is promised by the successful operation of the South's first "slab concentration yard" by Clancy Forest Products, Inc., at Decatur, Ala.

The heart of the new plant, located on the bank of the Tennessee River, is a hydraulic slab debarker designed by President Leon Clancy himself and constructed by Jackson Industries, Birmingham, Ala.

The efficiency of the machine enables Clancy to pay $8.00 a cord for barky slabs and edgings delivered to his yard. That's sufficient incentive for mills as much as 75 miles away to offer their full production of pine slabs. Available slab supply will more than meet the needs of his weekly production of 50 cords of chips.

The Clancy-designed slab debarker features two major differences from previously available equipment.

Its 900 p.s.i. water jets (originally there were two, but a third has been added) strike the barky surface of the slabs at a 30° angle, producing slabs that are absolutely bark-free, even around knots, with no damage at all to the wood itself. Heretofore, jets have been directed at a 90° angle to the slabs, resulting in considerable wood damage and relative loss of fiber.

Another new feature of the machine is powerful hold-downs—top and bottom rolls on both infeed and outfeed sides—which hold slabs in a steady position while passing through the force of the jets. This, too prevents destruction of wood while improving efficiency of bark removal.

The Clancy unit, costing about $20,000, also represents a

considerably lower investment than required by equipment previously available.

Mr. Clancy is making the unit available to the industry, and other individuals are already considering the installation of slab concentration yards. It isn't inconceivable that some day every mill in the South will be within reach of a slab processing plant.

At a recovery rate of one-half cord of chips per thousand feet of lumber, such a millenium in this new field of conservation could theoretically provide for a 40% reduction in the region's annual pulpwood cut or, conversely, a corresponding expansion in the paper industry without additional drain on the forests. In addition, it affords sawmills a means of recovering some $3.00 or more per thousand on their stumpage costs.

### Schundler

F. E. Schundler & Co. has completed plans for a new Perlite insulation board plant at Joliet, Ill. Composed principally of Perlite, this board is a research development of the Schundler laboratories, it was announced by J. C. Kingsbury, vice-president of the company. It culminates a long search for a mineral insulation in rigid board form. Both standard and acoustical board, in thickness from 1/4 to 2 in., will be produced at the new location. Present plans call for an output of 50,000 sq. ft. of 1-in. board per 24 hr. of operation, but the mill has been designed for a capacity of 100,000 sq. ft. Johnson & Johnson, Chicago, Ill., are the engineers for the project.

The complete stock preparation system and the board machine will be built by Downingtown Manufacturing Co., Downingtown, Pa. Equipment for the drying section will be supplied by Coe Manufacturing Co., Painesville, Ohio.

Stock will be prepared in a 2B1 Downingtown Fibrepulper and the board will be formed on a 105-in. wide specially designed fourdrinier. A unique feature of the installation will be a welded steel blow box positioned inside of the top wire just before the caliper press and just over the second of two Rotabelts. The blow box is expected to improve operation by heating the water in the sheet for more complete removal.

Drying equipment will include a Coe board drier and wet saw.

Production from the new plant will round out the Schundler line of fine insulation products and will integrate the use of a considerable quantity of Perlite from the company's mine.

The spring of 1955 has been set as the starting goal for the mill.

### Rome Kraft

Top administrative and production positions have been filled at the Rome Kraft Co., Rome, Ga. The big new plant, off the Alabama Road just west of Rome, is owned jointly by the Mead Corp. and Inland Container Corp. It is costing in excess of 25 million dollars.

Al H. Mahrt is president of Rome Kraft. A vice-president and director of the Mead Corp., he has had many years in the paper industry. Supervisory personnel of Rome Kraft includes veterans of many years in the papermaking industry, men who have learned their jobs from the ground floor up, and some newcomers who had never seen the inside of a paper mill before. Some of the supervisors were transferred here from other plants, and some are local men.

Herbert A. Kidd, vice-president and general manager of Rome Kraft, is a native of Scotland, and joined the Mead organization in 1929. He held successively important positions, and went to Rome from Macon, where he directed the operation of Macon Kraft Co. Mr. Kidd is a vice-president of the Mead Corp., and he continues as vice-president and general manager of Macon Kraft Co.

Top assistant to Mr. Kidd is Lawrence C. Crowder, general superintendent. Mr. Crowder was transferred from Macon Kraft, where he was plant engineer. He has been in Rome since the start of construction of the local plant. A native of Louisiana, he worked for paper mills in that state before joining the Macon Kraft organization.

Another of the first officials on the scene was E. V. McSwiney, assistant controller, who was transferred from Macon Kraft to Rome. Robert J. Kelly is industrial relations manager for Rome Kraft. He has held assignments at Chillicothe, Ohio, Escanaba, Mich., Kingsport, Tenn., and Sylva, N. C., before his transfer to Rome. He is a native of Springfield, Ohio. D. F. Black, Jr., native of Decatur, Ga., and graduate of Georgia Tech., is assistant industrial relations manager. He worked at plants in Chillicothe, Lynchburg, Va., and at Macon Kraft before his transfer. Ralph J. Schmieder is purchasing agent of Rome Kraft. He is a native of Ohio, and worked at Chillicothe and Macon before moving to Rome. Marvin O. Small, who worked in Chillicothe for 12 years, is senior accountant and office manager. He is a native of Chillicothe.

Marvin C. Jones, native of Trion, has been appointed supervisor of safety at Rome Kraft. A graduate of the University of Georgia, he was an instructor there and at the Off Campus Center in Rome before joining Rome Kraft this Summer. Anthony J. Parrino is superintendent of the paper mill. As such, he heads that part of the plant where the container board actually will be made. He worked for International Paper Co. before joining Macon Kraft six years ago. He is a native of Louisiana. James A. Whelan, also formerly with Macon Kraft, heads the pulp mill section of the plant. Under his supervision is the processing of pine logs into pure cellulose pulp. Also a native of Louisiana, he was at Macon Kraft six years. He worked for several other paper mills before joining Mead. James Foster Quinn, native of Alabama and graduate of the University of Alabama, is wood supervisor, and is in charge of woodyard operations at the plant. He was previously with the St. Joe Paper Co. Plant engineer is George W. Reynolds who transferred to Rome after three years at Macon Kraft. He was at Chillicothe two years before that. He is a native of Ohio and a graduate of the University of Illinois. Engineer is Kenneth Alfred Riddle, a native of Alabama and graduate of Auburn. He formerly worked for the Westinghouse Electric Corp. Another Alabamian, McDewain Sandlin, is chief chemist for Rome Kraft. He is a graduate of the University of Alabama and has had

***Top administrative officials of the new Rome Kraft plant are (seated, left to right) R. J. Kelly, industrial relations manager; E. V. McSwiney, assistant controller; Herbert A. Kidd, vice president and general manager; R. J. Schmieder, purchasing agent; N. R. Harding, woodlands manager; (standing) D. F. Black, Jr., assistant industrial relations manager; M. O. Small, senior accountant and office manager; and H. B. Mathias, wood procurement supervisor***

***Production supervisors at Rome Kraft are (left to right, seated) A. D. Simpson, power superintendent; A. J. Parrino, paper mill superintendent; L. C. Crowder, general superintendent; J. A. Whelan, pulp mill superintendent; D. H. Jones, master mechanic; (standing) C. W. Reynolds, chief engineer; M. D. Sandlin, chief chemist; E. W. Clark, chief meter man; J. F. Quinn, wood supervisor; and J. S. Kirkland, chief electrician***

paper mill experience. William E. Ross, native of Young Harris Ga., is assistant chief chemist. He is a graduate of Mercer University, and worked for Macon Kraft six and a half years. John S. Kirkland, veteran of six years' service at Macon Kraft, is chief electrician for Rome Kraft. A native of Pearson, Ga., he worked at paper mills in Georgia and Florida before joining the Macon Kraft organization. David H. Jones was promoted from pipefitter foreman at Macon Kraft to the position of master mechanic at Rome. He was at Macon six years, and is a native of Douglas, Ga. Albert D. Simpson is power superintendent at Rome Kraft, a position he also held at Macon where he was employed six years. He is a native of Norfolk, Va. Emory W. Clark is chief meter man. He is a native of Jacksonville, Ga., and is a graduate of Georgia Tech. E. M. Wiggins is millwright foreman. He worked for three other paper mills before joining the Rome organization. He is a native of Pearson, Ga. Building and grounds foreman is Newton E. Wagner, native of Rome. He formerly worked at Celanese and with the du Pont Co. Chalmers L. Keith is machinist foreman. He is a native of Geneva, Ala. He was with Macon Kraft before coming to Rome. Pipefitter foreman is Willian A. Snellgrove, native of Dothan, Ala. He has had extensive experience in paper mill construction. Tour foremen in the paper mill are James Oliver Edwards, who was at Macon six years; Orville W. Harris, who worked at paper mills in Savannah and St. Marys, Ga., and in Arkansas; and Roy F. Gaskin, who came here from the St. Joe Paper Co. in Florida. Tour foremen in the pulp mill all have had extensive experience at other plants. They are Murphy J. Brown, O. W. Padgett, and Alton Branch, each of whom was at Macon Kraft six years; and Dudley Grantham, at Macon and Coosa River Newsprint before transferring to Rome. John B. Oxford, Jr., formerly at Macon Kraft, heads the finishing and shipping department. He is a native of Macon.

N. R. Harding is woodlands manager of Rome Kraft. He joined the Mead organization at Macon in 1947, after working as a district forester in his native state of Florida and service with International Paper Co. He is a graduate of Penn State Forest School and North Carolina State College. H. B. Mathias is wood procurement supervisor. A native of Georgia, he worked for St. Joe Paper Co. before joining Macon Kraft eight years ago. C. A. Rodgers, formerly at Macon Kraft, is assistant wood procurement supervisor. He is a native of Ellijay, Ga., and is a graduate of the University of Georgia.

### Packaging Shows

Britain's Fourth International Packaging Exhibition, to be held at Olympia, London, Jan. 18–28, 1955, will be the largest and most comprehensive yet staged. The exhibition will show the latest means that have been developed to overcome all kinds of packaging problems.

The Third Canadian National Packaging Exposition will be held in Toronto at the C.N.E. Automotive Building, Nov. 9–11, 1954.

### Chem Show

The Eighth National Chemical Exposition will be at the Chicago Coliseum, Oct. 12–15, 1954.

### Achema XI

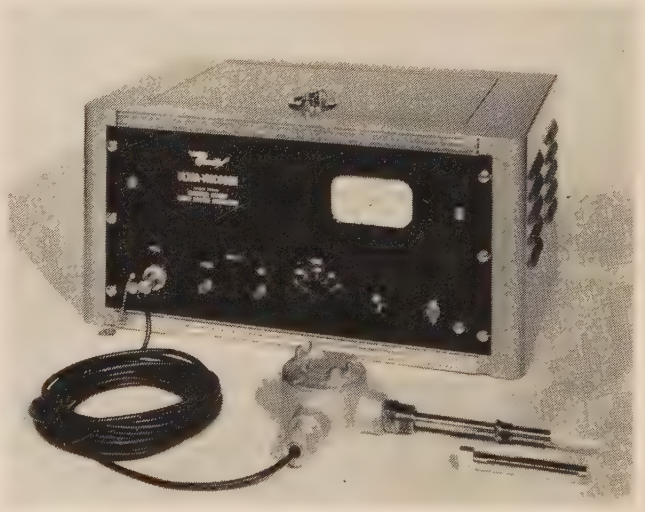
Achema XI—Chemical Apparatus and Equipment Congress and Exhibition, will be held in Frankfurt am Main May 14–22, 1955. Descriptive pamphlets are available from Dechema, Frankfurt am Main-W 13, Germany.

### NBS

Irl C. Schoonover has been appointed chief of the Mineral Products Div. of the National Bureau of Standards, succeeding Herbert Insley who retired recently. William D. Appel has been named assistant chief of the Organic and Fibrous Materials Div., and Norman Bekkdahl, chief of the Polymer Structure Section.

### Bendix

Bendix Aviation Corp., 203 W. 3rd St., Cincinnati 2, Ohio, is manufacturing and marketing the Bendix Ultra-Viscoson,



***Bendix Ultra-Viscoson***

an ultrasonic viscometer for controlling the color shade of gravure inks by controlling the ink viscosity.

### Patton

Patton Mfg. Co., Springfield, Ohio, has added a paper mill div. to its plant, for the development of special machinery and accessories for the paper industry. George S. Herbert, formerly assistant general manager and assistant treasurer of Midwest Fulton Machine Co., Dayton, Ohio, is general manager of the new division. Sales manager is Wayne T. Crannell, formerly chief draftsman for Midwest Fulton.

### Inco

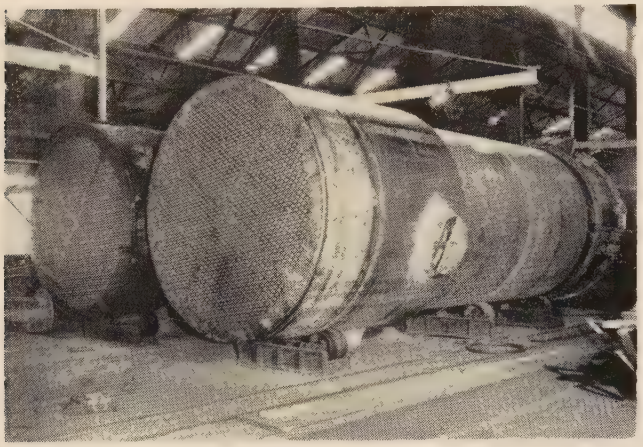
International Nickel Co., 67 Wall St., New York 5, N. Y., has published a 36-page glossary of terms for use by producers and users of ferrous and nonferrous castings.

### Hygrotester

Hygrotester, Inc., Brooklyn 1, N. Y., has issued a pamphlet describing the "Hygrotester-Boy," a portable dielectric moisture measuring instrument for pulp, paper, board, cellophane, etc.

### Goslin-Birmingham

Goslin-Birmingham Mfg. Co., Birmingham, Ala., has completed six 30-ft. heating elements designed to evaporate 500,000 lb. of water per hour in the recovery system of Champion Paper and Fibre Co., Canton, N. C.



***Two of the six elements at the Goslin-Birmingham Mfg. Co. plant prior to shipment***

### Reliance

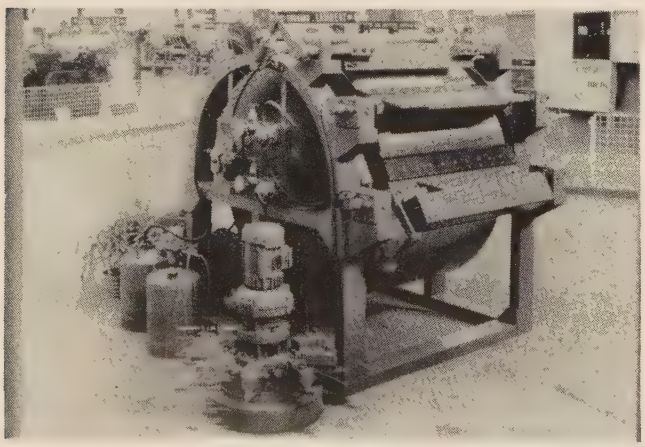
Reliance Electric and Engineering Co., Cleveland, Ohio, has published a booklet, "The Tools of Automation," expressing the company's philosophy for providing automation of single machines or continuous processes.

### R & I

Refractory & Insulation Corp., 120 Wall St., New York, N. Y., has issued a technical report (No. 54-2) on R & I Chromio for recovery boilers of paper mills.

### Lodding

Lodding Engineering Corp., Worcester, Mass., made a licensing arrangement with E. & M. Lamort Fils of Vitry-le-Francois (Marne), France, for the manufacture of Lodding doctors, blade holders, blades and oscillating equipment for the European paper industry, a year ago. Results of this collaboration were exhibited at the Fourth International Exposition of Paper and Graphic Techniques at the Grand Palais in Paris from May 1 to 12, when several different types of Lodding oscillating doctors built in France by E. & M. Lamort Fils were exhibited in operation.



***E. & M. Lamort Fils exhibit at the Paris Exposition***

### Olin Mathieson

The merger of Olin Industries, Inc. and Mathieson Chemical Corp. to form Olin Mathieson Chemical Corp. became effective August 31. John M. Olin is chairman of the new company and Thomas S. Nichols is president.

### B & W

The Babcock & Wilcox Co., Tubular Products Div., Beaver Falls, Pa., has issued a data card (TDC 147) titled "Helpful Hints on Hand Threading Stainless Steel Pipe and Tubing."

B & W has engaged in atomic energy work since the days of the Manhattan Project, and established an Atomic Energy Div. in April, 1953. This division is now staffed by more than 50 engineers and scientists engaged in nuclear design and development work, in preparation for the entry of private enterprise into the atomic power business.

G. H. Weight has been appointed southwest district sales manager for the B & W Tubular Products Div.

### Cyanamid

The Paper Chemicals Dept. of American Cyanamid Co. has opened a new development and applications laboratory in Chicago. The new laboratory will work with mill customers to experiment with Cyanamid's newer paper chemicals as they may be used in pulp furnishes.

Cyanamid's Research Div. has formed two new departments, the basic Research Dept. and the Research Service Dept., both at the Stamford, Conn., research laboratories. D. J. Salley has been named assistant director of the Basic Research Dept., of which J. T. Thurston, director of Stamford Research Laboratories, is acting director. R. H. Kienle has been appointed director of the Research Service Dept. with R. P. Chapman as assistant director.

### Raybestos-Manhattan

Frank A. McBrearity has been appointed Philadelphia branch manager of the Industrial Rubber Products Div. of Raybestos-Manhattan, Inc., Passaic, N. J. Howard W. Smith succeeds Mr. McBrearity as sales representative for the central New York area.

### Stebbins

The Stebbins Engineering and Manufacturing Co., Watertown, N. Y., has started construction of a new building to provide warehouse facilities near Pensacola, Fla.

### Virginia Smelting

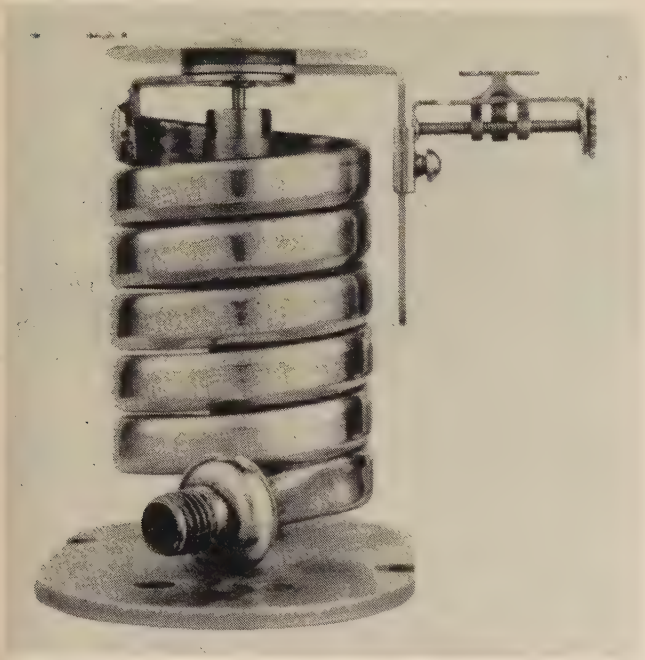
Jesse A. Weatherford has been appointed manager of the Industrial Sales Dept. of the Virginia Smelting Co., West Norfolk, Va. D. W. Duncan has been promoted to plant manager.

### J-M

Johns-Manville, 22 E. 40th St., New York, N. Y., has issued three descriptive sample folders to introduce a new series of composite asbestos electrical insulations. Folder EL-54A covers Quinterra Type 3-GR, a class H electrical insulation for temperatures up to 250°C. Folder EL-49A covers Quinterra Type 5-GR and G-GL, class B purified asbestos electrical insulations for temperatures up to 150°C. Folder EL-55A covers Quinorgo No-4000-GR and No-4000-GL, economical class B purified asbestos insulations. Another new J-M pamphlet, "Chempac Packings & Gaskets," describes several types of Teflon-treated asbestos packings.

FOXBORO

The Foxboro Co., Foxboro, Mass., has published a bulletin



*New Foxboro type 240 helical element for pressure measurement*

(6-10) describing a new line of helical pressure-measuring elements.

DOW

The Dow Chemical Co., Midland, Mich., has begun construction of new biochemical research laboratories at Midland. Also, a new latex research laboratory is expected to be occupied by November 1, and two other buildings, to be devoted to latex product development, will be completed in 1955.

FIBERGLAS

Owens-Corning Fiberglas Corp., Toledo, Ohio, has appointed 57 distributor-contractors of Kaylo industrial heat insulations which are effective to 1200°F.

Marshall D. Burch has been appointed director of industrial and personnel relations of the company. Fred R. Coumbe has been named manager of the Kansas City, Kan., plant, and Howard B. Richter is now manager of the Newark, Ohio, plant.

HOOKER

Harris C. Miller has been appointed New York district sales manager for Hooker Electrochemical Co., Niagara Falls, N. Y.

Hooker has purchased a barge for transporting liquid caustic soda in bulk to customers on the inland waterway system in the Chicago area.

G.E.

General Electric Co., Schenectady, N. Y., is producing a new process timer, the TSA-21, featuring "one-knob" control. The new timer will control the timed simultaneous opening of one electric curcuit and closing of another.

Another new G.E. product is a line of d.c. magnet brakes featuring reduced maintenance, easy single-point adjustment, and long life.

A new G.E. bulletin (GED-1966 A) gives information on electrical equipment for process industries.

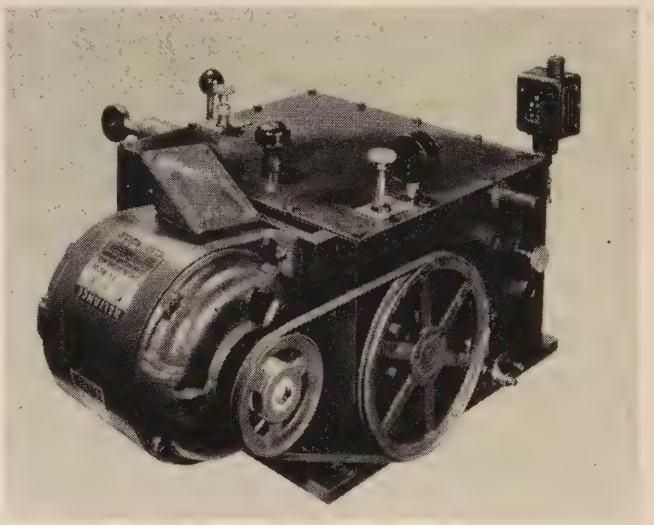
RHODIA

H. C. Nichols has been appointed head of the Engineering Service Section of the Industrial Reodorants ("Alamask") Div. of Rhodia, Inc., 230 Park Ave., New York, N. Y.

Rhodia is now offering specially designed reodorants for airborne odor abatement from sulphate pulp mill recovery operations.

ROSS MIDWEST-FULTON

Ross Midwest-Fulton Corp., Dayton, Ohio, has developed a rewinder attachment, the Hydroscillator, which imparts



*Ross Midwest-Fulton Corp. Hydroscillator*

oscillations to the rewinder to prevent wrinkles in the oncoming web and avoid the formation of hard and soft spots in the roll.

FARRAND

Farrand Optical Co., Bronx Blvd. & E. 238th St., New York 70, N. Y., has issued a bulletin (No. 818) describing the Farrand interference microscope for measuring surface roughness and finish.

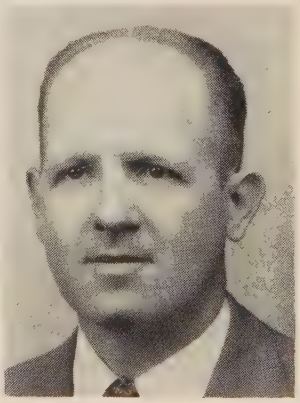
BAUER

F. A. Clayton has been appointed representative of the Bauer Bros. Co., Springfield, Ohio, for eastern Quebec, New Brunswick, Newfoundland, and Nova Scotia.

B-C

W. A. White, Jr., has been named vice-president of Black-Clawson Co. and general manager of the newly acquired Bagley & Sewall Div. of B-C at Watertown, N. Y. The new division will continue to manufacture high-speed fourdrinier machines.

*F. A. Clayton, Bauer Bros. Co.*



*W. A. White, Jr., the Black-Clawson Co.*

GURLEY

W. & L. E. Gurley, Troy, N. Y., has issued a bulletin describing the new RD Smoothness Tester for determining the smoothness of both felt and wire sides of the paper separately and simultaneously.

*Gurley RD smoothness tester*

BRANSON

Branson Instruments, Inc., Stamford, Conn., is manufacturing a new, heavy-duty, 2-kw. ultrasonic power generator, the Sonogen 2000, for speeding up the cleaning and degreasing of parts made of metal, rubber, plastics, glass, ceramics, etc.

MONSANTO

Desmond B. Hosmer has been named manager of the Anniston, Ala., plant of Monsanto Chemical Co.'s Organic Chemicals Div. Wallace K. Belin, formerly Anniston plant manager, is to become production manager of the Monsanto plant now under construction at Kearny, N. J.

SPROUT, WALDRON

Sprout, Waldron & Co., Muncy, Pa., are exclusive distributors of the Harvestore, a glass-surfaced steel storage structure manufactured by A. O. Smith Corp. The new storage structure is made of steel plate to both sides of which a layer of glass is fused.

NOPCO

The Paper Chemicals Div. of Nopco Chemical Co., Harrison, N. J., has developed an automatic and completely mechanical method for accurately measured feeding of additives of all types to the pulp and paper system. The new technique is known as the Acumeter Synchronous Flow System.

Nopco has introduced a new series of polyvinyl acetate emulsions for adhesives, paper, and textiles. The new line was featured at a recent Nopco industrial sales meeting.

*At the Nopco Industrial Sales meeting, front row, left to right: L. E. Rossiter, T. V. Rankin, B. A. Schiller, W. B. Morehouse, W. E. Brewer; second row: O. E. Lohrke, E. A. Robinson, R. F. Spain, J. N. Gammon, H. A. Swanson, R. B. Macintyre*

GOULDS

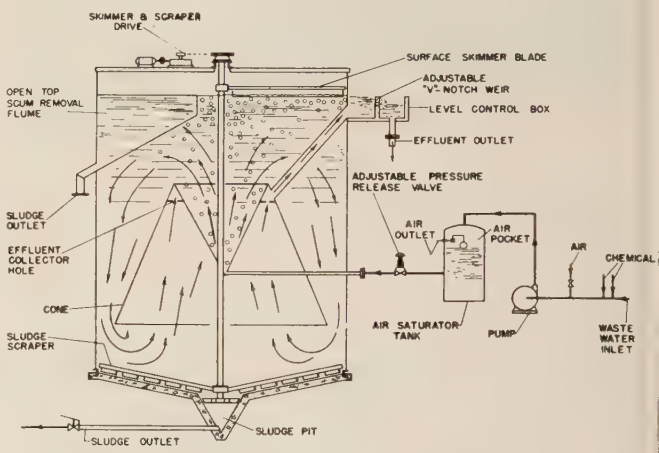
Goulds Pumps, Inc., Dept. TA, Seneca Falls, N. Y., has issued a bulletin (725.4) covering its new Fig. 3715 line of centrifugal pumps designed especially to provide dependable pumping of acids, alkaline liquors, slurries, and other process liquids.

B & O

On July 20 the Baltimore and Ohio Railroad started its "Tofcee," trailer-on-flatcar service. Specially designed flatcars carry two truck trailers, which are moved to and from terminals by tractors. B & O feels that the new service will provide shippers with fast door-to-door service on precise schedule, and at the same time will remove heavy freight from the overcrowded highways. Tofcee is in effect between Philadelphia and Baltimore, Pittsburgh and Chicago, and Cincinnati and East St. Louis

GRAVER

Graver Water Conditioning Co., 216 W. 14th St., New York, N. Y., is offering a new flotation unit, the Graver Aero-




*Sketch of the Graver Aeroflotor*

flotor for use in the pulp and paper, metallurgical, chemical, and petroleum fields. The new unit is capable of handling both floatable and settleable particles at the same time.

DU PONT

The peroxygen products sales development group of du Pont Co.'s Electrochemicals Dept. has been moved from Niagara Falls, N. Y., to Wilmington, Del. Included in the group are Joseph S. Reichert, Neil J. Salter, Lewis W. Blight, Francis LeRoy Fennell, Paul E. Kiefer, George E. Smedberg, and John G. Wallace.

du Pont is planning a $2,550,000 laboratory building at Wilmington for long-range and fundamental research by the Electrochemicals and Pigment Depts.

C.E.

Warren L. MacDonald has been appointed district manager of the Los Angeles office of Combustion Engineering, Inc., New York, N. Y.

EGAN

Frank W. Egan & Co., Bound Brook, N. J., have appointed ER-WE-PA, Erkrath, bei Dusseldorf, Germany, as their licensee to build paper converting and plastic equipment.

DIAMOND ALKALI

George Rieger, of Wilmington, Del., has been appointed by Diamond Alkali Co., Cleveland, Ohio, to lead and coordinate

*J. J. Tumpeer, Witco Chemical Co.*

*S. J. Briscoe, Pusey & Jones Corp.*

market research activities of its new Commercial Development Dept.

WITCO

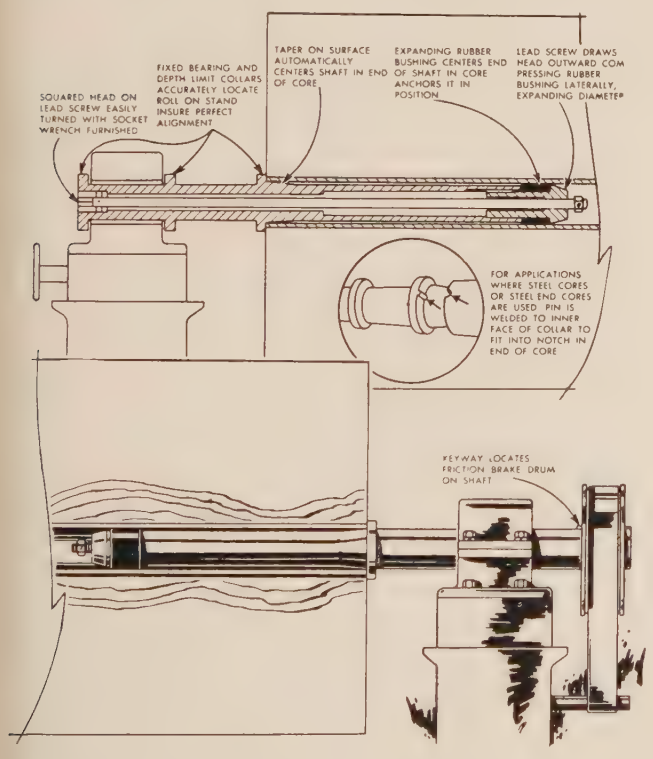
Joseph J. Tumpeer has been appointed senior vice-president of Witco Chemical Co., 260 Madison Ave., New York, N. Y.

P & J

Sidney J. Briscoe, New England district sales representative for the Pusey and Jones Corp., has moved to 93 LeMay St., West Hartford, Conn. His telephone number is Jackson 3-0013.

CLARK-AIKEN

The Clark-Aiken Co., Lee, Mass., is manufacturing a new stub shaft assembly said to greatly facilitate and reduce the


SQUARED HEAD ON LEAD SCREW EASILY TURNED WITH SOCKET WRENCH FURNISHED
FIXED BEARING AND DEPTH LIMIT COLLARS ACCURATELY LOCATE ROLL ON STAND INSURE PERFECT ALIGNMENT
TAPER ON SURFACE AUTOMATICALLY CENTERS SHAFT IN END OF CORE
EXPANDING RUBBER BUSHING CENTERS END OF SHAFT IN CORE ANCHORS IT IN POSITION
LEAD SCREW DRAWS HEAD OUTWARD COMPRESSING RUBBER BUSHING LATERALLY, EXPANDING DIAMETER
FOR APPLICATIONS WHERE STEEL CORES OR STEEL END CORES ARE USED PIN IS WELDED TO INNER FACE OF COLLAR TO FIT INTO NOTCH IN END OF CORE
KEYWAY LOCATES FRICTION BRAKE DRUM ON SHAFT

labor involved in making roll changes on all types of equipment in which paper, board, film, foil, or fabric is fed from rolls on roll stands.

CLEAVER-BROOKS

Cleaver-Brooks Co., Milwaukee, Wis., has issued a bulletin (AD-135) describing the new CB50–80 line of small boilers.

HONEYWELL

Minneapolis-Honeywell Regulator Co., Philadelphia, Pa., has published a bulletin (100-C) listing all current Honeywell Industrial Div. literature.

GENERAL MILLS

General Mills, Kenedy, Tex., is producing galactomannan gum from the guar bean by a process which was developed by the company. The gum is used to increase mullen and tear and to improve clay retention.

GENERAL AMERICAN TRANSPORTATION

The Fuller Co., Catasauqua, Pa., has been acquired by General American Transportation Corp., Chicago, Ill. Fuller manufactures pneumatic conveying systems for dry bulk materials handling.

A-C

Allis-Chalmers Mfg. Co., Milwaukee, Wis., has issued a bulletin (28 × 8155) giving the mineral name, chemical formula, usual areas in which deposits are found, the normal causes, and suggested preventive measures for deposits found in steam-water cycles.

CARBORUNDUM

I. Wendell Hamm of Seaford, Del., has been appointed director of manufacturing for the Carborundum Co., Niagara Falls, N. Y.

LINK-BELT

Link-Belt Co., 307 N. Michigan Ave., Chicago, Ill., has issued a new 340-page standard products catalog 950.

KAOLIN

A Georgia court recently ruled that a processing patent owned by Georgia Kaolin Co. is good and valid, but that Thiele Kaolin Co. is not violating it, as had been charged by Georgia Kaolin.

# OBITUARY

## Geoffrey Broughton

Geoffrey Broughton, chairman of the Department of Chemical Engineering, University of Rochester, Rochester, N. Y., died on Sept. 10, 1954, in the Strong Memorial Hospital, Rochester, N. Y.

Mr. Broughton was born in Rockdale, England, on June 13, 1912, and received his Sc.D. degree in chemical engineering from the Massachusetts Institute of Technology in 1938.

He joined the Eastman Kodak Co., Rochester, N. Y., as a chemical engineer in 1938. In 1945 he became assistant superintendent of the Paper Service Department. From 1943 to 1948 he was liaison officer in the Office of Scientific Research and Development in Washington, D. C.

From 1949 to 1952 he was head of the Pulp and Paper Department of the Lowell Textile Institute, Lowell, Mass. He joined the staff of the Chemical Engineering Department of the University of Rochester in 1952, becoming chairman of the department in 1954.

Mr. Broughton published many scientific papers and was an active participant in the work of the Chemical Methods Committee of the Technical Association of the Pulp and Paper Industry.

# TAPPI DIVISIONS AND COMMITTEES

*Reports of Activities*

## *Structural Fibrous Materials Committee*

*Main Committee*

V. M. Anderson, Fir Tex Insulation Board Co.
H. L. Becker, Homasote Co.
J. F. Campbell, Flintkote Co.
A. H. Chamberlain, Bird & Sons, Inc.
G. H. Chidester, Forest Products Laboratory
E. Frederick, Jr., Celotex Corp.
O. W. Frost, Forest Fiber Products Co.
D. L. Gleaves, National Gypsum Co.
E. M. Jenkins, Johns-Manville Corp.
W. D. MacKinnon, Donnacona Paper Co., Ltd.
W. J. Marshall, Insulation Board Institute
T. R. Naffziger, Northern Regional Research Lab.
J. J. Perot, Flintkote Co.
W. L. Scott, Armstrong Cork Co.
R. J. Shouvlin, Bauer Brothers Co.
G. W. Thomas, Wood Conversion Co.
R. S. von Hazmburg, U. S. Gypsum Co.
A. T. Walton, Simpson Logging Co.

*Subcommittee Chairmen and Members*

E. M. Cagle, Flintkote Co.
H. B. Carson, Fir Tex Insulation Board Co.
G. E. Chamberlain, Minnesota & Ontario Paper Co.
L. Eberhardt, Bauer Brothers Co.
J. J. Erchul, Pioneer Flintkote Co.
W. H. Graham, Johns-Manville Corp.
C. E. Hrubesky, Forest Products Laboratory
F. R. Hyatt, Johns-Manville Corp.
E. J. Jones, Celotex Corp.
E. A. Lauring, Minnesota & Ontario Paper Co.
Wayne C. Lewis, Forest Products Laboratory
M. C. Miller, Johns-Manville Corp.
R. H. Neisel, Johns-Manville Corp.
E. J. Reichman, Simpson Logging Co.
R. J. Siedl, Forest Products Laboratory
H. M. Sutcliffe, Celotex Corp.
R. D. Ziegler, University of Washington

The following reports have been received since the May report.

*S-0. Steering Committee.* G. W. Thomas of the Wood Conversion Co. has been appointed to the Steering Committee.

*S-1. New Projects and Reviews.* New assignments are being considered for those subcommittees who will finalize their present projects during 1954: S-5, S-9, and S-10.

*S-2. Papers, Publications, and Reviews.* Nothing to report.

*S-3. Test Board Formation.* Board-forming equipment evaluation has been completed by the Celotex Corp. Results are being evaluated at present. Board mold assembly is to be dismantled and shipped to another member for evaluation.

*S-4. Drainage Time Testing.* The two S.F.M.C. drainage time testers are now being evaluated by the Flintkote Co. at Meridian, Miss., and the Insulite Division of M&O Paper Co. at International Falls, Minn.

*S-5. Transverse and Deflection Testing.* The final draft of "Flexure Test for Insulating Fiberboard, T 10003 sm-53" has been submitted to members of the main committee for approval.

*S-6. Water Absorption, Water Penetration, and Water Vapor Permeability.* No report.

*S-7. Nail Holding and Racking Test.* A proposed course of action for a laboratory study of stress-strain analogies between the full-scale racking test and small-scale tests which measure physical properties of insulation board has been reviewed by the committee and agreement reached for action. It is planned to secure proposals for handling this project from several qualified independent laboratories in the next few months.

*S-8. Light Reflection Measurement.* Samples of fiberboard having a matte finish and samples with a somewhat glossy finish have been received from Donnacona and the light reflectance of these samples has been determined by the G.E. spectrophotometer and the Hunter multipurpose unit. The samples, together with the above test data, have been sent to Miller of Johns-Manville for correlation studies with the Baumgartner Sphere.

*S-9. Consistency Measurements on Insulating Board Samples.* The final revision of a suggested standard procedure on consistency measurements has been completed and mailed to all subcommittee members.

*S-10. Test Boards vs. Handsheets for Board Pulp Evaluation.* Subcommittee has new assignment under consideration.

*Progress Report on the Development of a Small-Scale Tunnel for Measuring Rate of Flame Spread.* Design experimentation has been completed and a new small tunnel is under construction embodying all refinements that have been made to date. Upon completion, correlation work with the Underwriters' tunnel test will be undertaken.

J. J. Perot, *Secretary*

## *Plastics Committee*

### *Fibrous Materials for Laminated Products Subcommittee*

A meeting of the above subcommittee took place at TAPPI headquarters, 155 E. 44th St., New York 17, N. Y., on Sept. 15, 1954, at 10:00 a.m. The following attended:

Paul M. Goodloe, Brown Co., Chairman
V. A. Belcher, New Jersey Zinc Co.
R. H. Doughty, Fitchburg Paper Co.
W. E. Sanders, Panelyte Div., St. Regis Paper Co.
G. R. Shaw, Taylor Fibre Co.
F. B. Wardwell, Brown Co.
Ray Lodge, Borden Co.
B. R. Whaley, Sonoco Products Co.
D. S. Greif, National Starch Products
M. A. Hescock, Brown Co.
W. E. Morris, Barrett Div., Allied Chemical
E. Egan, Frank W. Egan Co.
V. T. Stannett, College of Forestry, Secretary

Apologies for absence were received from R. T. Nazzaro. The minutes of the previous meeting were read and approved. It was reported that 50 replies were received from the last minutes and of these 40 desired to participate in the activities of the Laminates Subcommittee and 10 asked to continue to receive the minutes and other correspondence relating to the work of the subcommittee.

The progress of the program for testing the resin saturation properties of paper was reported. After considerable discussion it was agreed that a detailed questionnaire would be sent out to members asking for specific information regarding the conditions and type of saturation test used, also the type of resin and paper recommended, together with the shortcomings of current test procedures. A further meeting will be held at TAPPI on November 5 to discuss the response to the questionnaire and arrange the laboratory work resulting from the questionnaire. It was hoped that all interested members, including the resin manufacturers, would attend.

The question of a program for the 1955 TAPPI Fall Plastics Conference (scheduled to be held in New York) was discussed. It was agreed that a half-day or one-day symposium of papers of interest to laminators should be aimed at and that cooperation with the unorganized group of laminators would be welcomed.

A note of thanks to R. J. Seidl for his past services as chairman of the subcommittee was approved unanimously.

The next meeting of the subcommittee will be held at 10:00 a.m. on Thursday, November 4, at TAPPI, 155 E. 44th St., New York N. Y.

The meeting terminated at 12:30 p.m.

V. T. STANNETT, *Secretary*

## *Electrical Engineering Committee—Subcommittee on Chipper Drives*

A subcommittee meeting was held on Thursday, Aug. 12, 1954, at Bowaters Southern Paper Corp., Calhoun, Tenn. The members present included:

W. F. Hughes, J. E. Sirrine Co., Greeneville, S. C.
R. R. Baker, Westinghouse Electric Corp., Pittsburgh, Pa.
G. L. Oscarson, Electric Machinery Co., Minneapolis, Minn.
H. V. St. Clair, Carthage Machine Co., Carthage, N. Y.
A. D. Snipes, Westinghouse Electric Corp., Chattanooga, Tenn.
G. E. Shaad, General Electric Co., Schenectady, N. Y.
N. H. Mailhos, Brunswick Pulp & Paper Co., Brunswick, Ga.
M. J. Osborne, Bowaters Southern Paper Corp., Calhoun, Tenn.

***Subject: The Selection of Chipper Drives***

PHASE I

The meeting was opened by M. J. Osborne, chairman, who asked R. R. Baker to give a résumé of data accumulated since the last meeting of the committee, Feb. 16, 1954. (Mr. Baker's letter to M. J. Osborne, dated August 6, was quoted.)

Very good reports had been received from Rhinelander Paper Co., Halifax Paper Co., Kimberly-Clark Corp., and St. Joe Paper Co., for several species of wood. Arrangements have been made by Westinghouse for obtaining figures from Camp Manufacturing Co., which will be available for the next meeting. It was brought out that St. Regis Paper Co. at Tacoma is now buying chips. Mr. Osborne added Bowaters Southern data to the tabulation. The modified Montreal tabulations of data were presented by Mr. Baker.

It was stated that the files of this subcommittee will be turned over to the secretary of the Electrical Engineering Committee on completion of the work.

***Abstract of the Report***

The Electrical Engineering Committee of TAPPI appointed this subcommittee, and charged it with the responsibility of arriving at power requirements for chipping various species of wood and for developing nomographs, charts, and other tools which will be useful in the selection of the proper induction or synchronous motor drives.

It was determined that the body of the report should contain:

1. The number of mills contacted.
2. Power data.
3. How the committee recommend that chipper motors be selected.

The maximum chip length will require maximum horsepower. The motor should be large enough to do the most difficult job.

The maximum log diameter is determined by the spout diameter and is 2 in. less than the spout diameter: 24-in. spout, then 22-in. log = maximum diameter.

If the normal production is in cords per hour, what should be the production figure for a 1, 2, or 4-hr. period? In a

***Subcommittee on Chipper Drives, left to right: R. R. Baker, Westinghouse Electric Corp., Pittsburgh, Pa.; G. L. Oscarson, Electric Machinery Co., Minneapolis, Minn.; W. F. Hughes, J. E. Sirrine Co., Greenville, S. C.; Sue Sharp, Stenographer, Bowaters Southern Paper Corp., Calhoun, Tenn.; N. H. Mailhos, Brunswick Pulp & Paper Mills, Brunswick, Ga.; H. V. St. Clair, Carthage Machine Co., Carthage, N. Y.; G. E. Shaad, General Electric Co., Schenectady, N. Y.; and M. J. Osborne, Bowaters Southern Paper Corp., Calhoun, Tenn.***

1/2-hr. period, you can get temperature rise. With a service factor of 15%, extreme production requirements would probably carry past the service factor in one hour. There are going to be other times when the knives will be somewhat dull, or a combination of these two factors, production and dull knives, which will use up the service factor.

It was decided to set maximum production for a 1-hr. period.

Dryness of the wood has a considerable effect on the power constant. Green wood has a low power factor; wood 3 months old has a high power factor.

Group I of the subcommittee's recommendations includes species A-1, 2, 3, B-1, 2, 3, 4, C-1, 2, ranging in shear stress parallel to the grain 690 to 1610 p.s.i. There were 29 power readings for this group. The average, minimum, and maximum readings were as follows:

Average—6.9 hp.-hr./cord
Minimum—4.1 hp.-hr./cord
Maximum—9.7 hp.—hr./cord

The committee suggests that a TAPPI recommended practice of 7 hp.-hr. per cord be adopted to cover the requirements of this group.

Group II of our recommendations includes species D-1, 2, E-1, 2, 3, 5, commonly classified as hardwoods ranging in shear stress parallel to the grain of 1490 to 2100 p.s.i.

There were only five power readings for this group. The average, minimum and maximum readings for this group were as follows:

Average— 8.6 hp.-hr./cord
Minimum— 7.7 hp.-hr./cord
Maximum—10.5 hp.-hr./cord

The committee suggests that a TAPPI recommended practice of 10 hp.-hr./cord be adopted to cover the requirements of this group.

CLASSIFICATION BY MR. BAKER

| | | |
|---|---|---|
| A-1. Fir | B-1. Spruce | C-1. Larch |
| 2. Balsam | 2. Poplar | 2. Pine (Southern) |
| 3. Aspen | 3. Pine (Northern) | |
| | 4. Hemlock | |
| D-1. Gum | E-1. Oak | |
| 2. Maple | 2. Birch | |
| | 3. Hickory | |
| | 4. Locust | |

| A | B | C | D | E |
|---|---|---|---|---|
| 5.2 | 6.1 | 9.7 | 7.7 | 9.0 |
| 8.7 | 8.5 | 6.5 | 8.14 | 7.15 |
| | 8.3 | 8.1 | | |
| | 5.7 | 7.1 | | |
| | 5.9 | 8.7 | | |
| | 7.0 | 6. | | |
| | 5.6 | 5.4 | | |
| | 7.1 | 6.9 | | |
| | 4.1 | 7.0 | | |
| | 4.6 | 7.0 | | |
| | | 5.9 | | |
| | | 5.6 | | |
| | | 7.3 | | |

*Note:* 9.7 under "C" includes blower.

Mr. St. Clair of Carthage Machine Co. is to send Mr. Osborne lists of power constants that are in use by his company.

Mr. St. Clair stated that the back-grind on knives is the worst thing to do on a knife. The action of back-grind causes power factor to go up, breaking the end of the chip.

The committee agreed that there was too little information collected to date to break the species down into more than two groups. The committee should continue to try to get more and better data so the groups can be broken down.

Mr. Osborne suggested that the committee make recommendation to TAPPI for funds. The committee was asked if the study should be continued, and if it should be presented to TAPPI that a task force, or a laboratory, be appointed by TAPPI, and paid for by TAPPI funds, to go to other mills, or to make experiments to get the information that is needed. Mr. Osborne's opinion was that the study was worth while and that probably TAPPI would provide funds to continue study.

Mr. Baker agreed that the committee has a long way to go, but suggested that the study is sufficiently worth while to call upon the Association for money to further the study. Tests received so far indicate that laboratory work would be well in order to study the more important phases of this work. Excellent reports from the Kimberly-Clark Corp. showed that tests were made on log sizes which varied from 8 up to 16 in. of measured wood.

**Table I. Tabulation of Hp.-Hr./Cord for Various Wood Species**

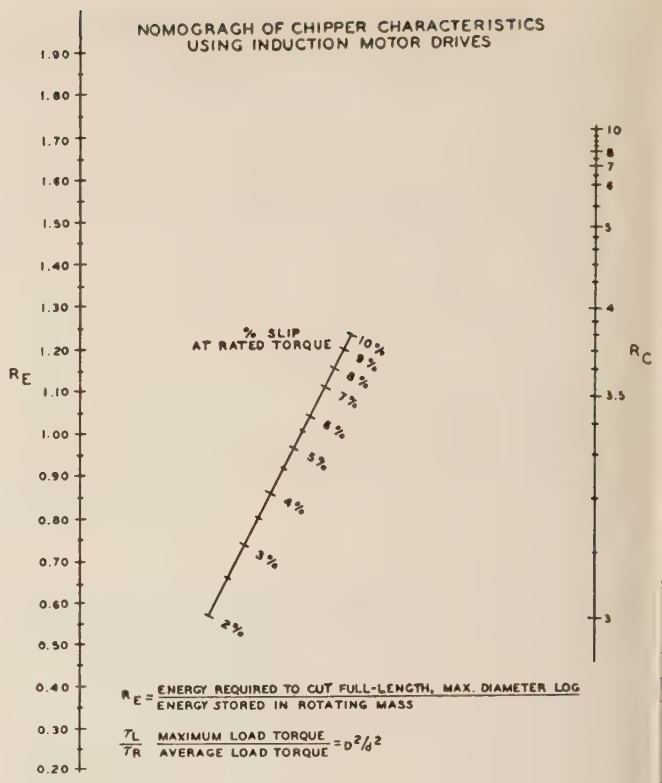
| *Species* | *Shear stress, p.s.i.* | *Recommended hp.-hr./cord* |
|---|---|---|
| Fir | 690 | 7 |
| Balsam | 690 | |
| Aspen | 800 | |
| Spruce | 950 | |
| Poplar | 980 | |
| Pine—Northern | 800/1040 | |
| Hemlock | 1010/1060 | |
| Larch | 1200 | |
| Pine—Southern | 1180/1350 | |
| Gum | 1490 | 10 |
| Maple | 1610 | |
| Oak | 1700 | |
| Birch | 1725 | |
| Hickory | 1870 | |
| Locust | 2100 | |

Mr. St. Clair drew a picture of a Murray chipper on the board: 39° angle, single grind has a straight axe action into the wood. This chipper breaks the chip less than 1/4 in. thick; frictional action will get a chip 1 in. thick. Murray will get a chip 1/4 to 3/4 in. thick.

Sumner-Murray: The knife is mounted on the surface of the disk. The reason for putting the knife through the disk is that it gives greater areas back of knife and longer life for knives.

Mr. Baker: Couldn't this be answered by laboratory work?

Mr. St. Clair: (Gave an explanation of the grinds of various knives.) In dry wood the power factor went high—changing


NOMOGRAGH OF CHIPPER CHARACTERISTICS
USING INDUCTION MOTOR DRIVES
% SLIP
AT RATED TORQUE
$R_E$
$R_C$
$R_E$ = ENERGY REQUIRED TO CUT FULL-LENGTH, MAX. DIAMETER LOG / ENERGY STORED IN ROTATING MASS
$\frac{T_L}{T_R}$ MAXIMUM LOAD TORQUE / AVERAGE LOAD TORQUE = $D^2/_{16}^2$

the knife angle did no good. Dry wood was very hard to handle. The energy factor depends on moisture—45 to 50% gives a cleaner chip.

Mr. Baker: Readings from Kimberly-Clark Corp.—average of 24 test readings, minimum was 4.3, maximum was 10.8 hp.-hr./cord. Grand average 7.1—increase in load—ran up 22% on the average above the double grind—chip log size 8 to 16 in. Power requirements went up as log diameter decreased. Heart wood is harder than outside.

Mr. Shaad: It takes more power to cut slab than round wood.

Mr. Baker: We should have enough data reported to us and should have enough money to get questions answered with laboratory tests.

Mr. Osborne: List of investigations for laboratory to make: effect of—

1. Knife grind.
   (a) Single bevel.
   (b) Double bevel.
   (c) Single bevel with back-grind.
2. Spout angle.
3. Species wood.
   (a) Effect of diameter.
   (b) Moisture content.
   (c) Seasonal variation.
   (d) Sectional variation.
4. Effect of chip length

If the chipper has a blower, the power for blowing should be figured separately.

PHASE II

Mr. Osborne put the formula on the board.

Mr. Baker: Discussion of formulas for synchronous and induction motors.

Mr. Shaad: Least expensive motor is 0.8 power factor motor with 250% pull-out torque.

Mr. Osborne: What we want is the most economical drive.

Recommendation: 0.8 power factor—250%.

Mr. Baker: Agreed.

Mr. Shaad: Agreed.

Committee recommendation: That 250% pull-out torque synchronous motors at 0.8 power factor be recommended.

Mr. Shaad: 90% of the time the buyer will get most for his money in the above recommendation.

Mr. Osborne: Any other cases should be treated as special. (Drawing on board.)

$NSD^2LK \times 0.4/3100$ = synchronous motor ratings.

Mr. Shaad: (Drawing on board.) Induction motor—250% BDT peak power demand—Item (IIE) divide by $R_{ci}$.

Mr. Osborne: We felt that it would not make very good chips to slow down to less than 75% synchronous speed.

Mr. St. Clair: Slowdown should be 20% maximum.

## Phase III

Mr. Osborne presented Mr. Shaad's nomographs.

Mr. Osborne asked Mr. Shaad and Mr. Baker to run cases through both ways.

(There was some difference in chart readings.)

Mr. Osborne suggested that the charts be checked and double checked and then recommend to TAPPI that they publish the data sheets.

Mr. Hughes: It would certainly be a help to people buying chippers and drives to get proper information.

Mr. Baker: Size of motor to apply—the committee has been working on this for three or four years.

Mr. St. Clair: Carthage Machine Co. has its own system based on practical experience. It is a graphical solution.

Mr. Osborne: I would like to have this third system check with the other two before presenting to TAPPI. Please send it down to us as soon as possible.

Mr. St. Clair: Chipper manufacturers would prefer a synchronous motor. (Will send charts within a month.)

Mr. Osborne: Is the committee in agreement to publishing both systems after checking?

Committee: Agreed.

Mr. St. Clair: Peak production is dependent on the way the chipper is fed.

Mr. Baker: Data are not too accurate. We should take these figures into account before sending any out.

Mr. St. Clair: Should we go through average and take a mean average and use that as motor selection?

Mr. Baker: That is one way of going about it.

Mr. St. Clair: Most people buy their chipper motor at about one-third actual chipper production.

Mr. Shaad: Maximum 1-hr. production is what summarizes what customer can get.

Mr. Hughes: How can one arrive at the average size of the logs?

Mr. St. Clair: Design the motor for large capacity.

Mr. Osborne: Mr. Oscarson, please look at the tabulation; figure from tabulation what is the satisfactory and accepted per cent of the maximum production. Would we make a radical departure to use 20% of the theoretical capacity? Brunswick mill is in trouble because of the chip blower.

Mr. St. Clair: I can get busy on my project right away. I believe we have data up to 84-in. chippers. The last time I looked at it, we had done 62, 74, 78, and 84-in. chippers.

Mr. Osborne: What should we recommend to use—belts or a direct drive for wound rotor motors?

Mr. Baker: The cost of drive motors makes a belt drive more satisfactory.

Mr. St. Clair: A V-belt is far more satisfactory in the long run.

Mr. Osborne: Chipper speeds are increasing.

Mr. St. Clair: Motor horsepower is going up—one should never put fly wheel on the disk. In the case of an induction driven motor, you can change it in one day. We are putting in two identical Norman chippers, one at Crossett with wound rotor V-belt drive and one at Rome with synchronous motor drive. We will be able to get a cost comparison soon.

Mr. Oscarson: What is the average life of V-belts on a 500-hp. drive?

Mr. St. Clair: About four or five years.

Mr. St. Clair: Energy requirements on the Norman would be around 8 to 8.5 hp.-hr./cord for pine. This is higher than for a standard chipper.

Note in recommendations: If the design of the chipper is such, then the seven that we are recommending is a little low.

Mr. Baker: The data we have include only the standard design chippers.

Mr. St. Clair: The Norman chipper has flat slope, Murray has even flatter slope; the flatter angle of knife does require more horsepower.

Recommendation: These data on which we have based our recommendations apply to standard chippers only; some of the more recent chipper designs may require more horsepower. This should be checked with the manufacturer. Tests are now being run which will determine these values and provide more information.

Mr. Osborne: We need to get full reports soon for the meeting, October 18, 19, and 20 at Philadelphia.

Mr. Oscarson: It would be good to check the three systems when we get them.

Mr. Osborne: You will check these three systems for us, will you not, Mr. Oscarson?

Mr. St. Clair: We are installing a rimless chipper (drew sketch on board)—running at 900 r.p.m.

Mr. Osborne: What kind of gear shift and drive will be used for this chipper?

Mr. St. Clair: International Harvester. The rimless chipper cuts like an axe—has compound angle of 32 to 38° on knife. Total of 72°—one half of that is 36°. The new chipper will revolutionize the chipper industry. Entirely different from normal design—will take less horsepower. Production figures are up to three times that of a standard chipper.

Mr. Baker: I have heard that the other chipper manufacturers are becoming interested in this committee work.

Mr. Shaad: I have heard the same.

Mr. Osborne: I tried to enlist aid for this work three years ago from Carthage, Murray, and Sumner. Carthage was the only company then interested in participating.

Mr. Baker: I suggest you send minutes of this meeting to Sumner and Murray with a note asking that they check our recommended power constants and tell them that we will appreciate their comments.

Mr. Osborne: I will do that. Mr. Shaad is to get nomographs here in two weeks. Mr. St. Clair is to get the charts here in two weeks to one month. I shall write to John Rich of Improved Machinery Co. for additional data—Impco is now making a chipper.

Meeting adjourned.

M. J. Osborne, *Chairman*

*Recommended Procedure for Chipper Drive Selection*

It was recommended that the mill engineers establish the following data:

I. Production:
   - A. Normal production_____cords/hr.
   - B. Maximum 1-hr. production_____cords/hr.

   Logs:
   - C. Wood species_____
   - D. Maximum log length____ft.
   - E. Average log diameter____in.

   Chipper:
   - F. Disk diameter____in.
   - G. Spout diameter____in.
   - H.* Total $WR^2$____lb. sq. ft.
   - J. Maximum speed chipper____r.p.m.
   - K. Number of chipper knives ___
   - L. Maximum chip length____in.
   - M. Method of removing chips from chipper

II. Calculate or establish the following:
   - A. (Hp.-hr./cord) for particular wood. Species from Table I

* Total of chipper disk, sheaves, or other rotating parts.

***Reading from left to right in the photograph of the group at work are: H. V. St. Clair (standing at board), G. E. Shaad, N. H. Mailhos, A. D. Snipes, M. J. Osborne, Mrs. Sue Sharp, W. F. Hughes, G. L. Oscarson, and R. R. Baker***

B. (Chipper feed) in ft./min.

$$= \frac{\text{chipper r.p.m.} \times \text{number knives} \times \text{chip length (in.)}}{12}$$

$$= \frac{\dagger IJ \times IK \times IL}{12}$$

C. Maximum permissible log diameter
= (spout diameter)—2 in.
= $D$
= $IG$—2 in.

D. (Maximum theoretical chipper capacity) in cords/hr.
= chipper feed $\times$ 0.00385 $\times$ $D^2$
= ‡(IIB) $\times$ 0.00385 $\times$ (IIC)$^2$

E. Peak power demand
= hp.-hr./cord $\times$ maximum theoretical capacity
= (IIA) $\times$ (IID)

F. Chipper capacity ratio = $R_c$

$$= \frac{\text{maximum theoretical capacity}}{\text{maximum 1-hr. production requirements}}$$

$$= \frac{\text{(IID)}}{\text{IB}}$$

G. Cords per log maximum permissible diameter full length

$$= \frac{\text{log length} \times D^2}{15{,}600}$$

$$= \frac{(ID) \times (\text{IIC})^2}{15{,}600}$$

H. Energy required to chip one log maximum diameter and full length in hp.-sec.
= hp.-hr./cord $\times$ cords/log $\times$ 3600
= (IIA) $\times$ (IIG) $\times$ 3600

J. Stored energy in chipper (in hp.-sec.)

$$= \frac{WR^2 \times (\text{r.p.m.})^2}{3.2 \times 10^6}$$

$$= \frac{(IH) \times (IJ)^2}{3.2 \times 10^6}$$

K. Energy ratio ($R_e$) to be used for induction motor

$$= \frac{\text{energy to chip maximum diameter full length log}}{\text{stored energy}}$$

$$= \frac{\text{(IIH)}}{\text{(IIJ)}}$$

III. Synchronous motor ratings:

A. Peak power demand when chipping log of maximum diameter $D$
= theoretical chipper capacity $\times$ hp.-hr./cord
= item (IIE)

B. Capacity ratio
= $R_c$ = item (IIF)

$$= \frac{\text{maximum theoretical capacity}}{\text{maximum 1-hr. production requirements}}$$

C. Where $R_c$
$\geqq$ 2.5 select synchronous motor rating

$$\geqq \frac{\text{item (IIE)}}{2.5}$$

D. Where $R_c < 2.5$ select synchronous motor rating
$\leqq$ hp-hr./cord $\times$ maximum 1-hr. production
= (IIA) $\times$ (IB)

IV. Induction motor ratings:

A. Establish energy ratio $R_e$

$$= \frac{\text{work}}{\text{stored energy}}$$

= item (IIK)

B. Establish capacity ratio $R_c$
= item (IIF)

C. From nomograph establish value of slip corresponding to the established values of $R_e$ and $R_c$

D. Where slip is 5% or under, select wound rotor motor as follows:
= hp.-hr./cord $\times$ maximum 1-hr. production
= IIA $\times$ IB

E. Where slip is 2% or under, select squirrel cage, wound rotor or synchronous motor with rating
= hp.-hr./cord $\times$ maximum 1-hr. production
= IIA $\times$ IB

F. Where slip exceeds 5%, select wound rotor induction motor rating basis of a value of $(R_c)_1$ from nomograph equivalent to 5% slip for established value of $R_e$ with rating

$$= \frac{\text{peak power demand}}{(R_c)_1}$$

$$= \frac{\text{item (IIE)}}{(R_c)_1}$$

† I refers to Section I of Procedure.
‡ II refers to Section II of Procedure.

## *Corrugated Containers Committee*

The minutes of TAPPI Corrugated Containers Committee held in New York City on June 18, 1954 are as follows. Those present were:

C. E. Jakeway and S. Stacy, American Box Board Co.
F. D. Long, Container Corp. of America
J. J. Koenig and C. G. Hornor, Gaylord Container Corp.
L. K. Burnett and R. G. Hessidence, Ohio Boxboard Co.
J. Fuko, River Raisin Paper Co.
W. D. Arbuthnot and W. Brown, Robert Gair Co.
H. T. Scordas, T. R. Hamilton, R. W. Hutcheson, and R. W. Pfeil, Union Bag & Paper Co.

The meeting was held in the TAPPI conference room, and was called to order at 10:00 a.m. Mr. Arbuthnot and Mr. Burnett explained that the object of the meeting was twofold. First to give the corrugated machinery manufacturers an opportunity to present their latest engineering innovations and second, to screen the manufactures for topics and speakers for the February, 1955, meeting.

Duane Arbuthnot introduced the speakers.

The first speaker was Charles Nitche of Langston, who explained that his company considered the following items as relatively new in corrugated machinery.

1. Hydraulic loading of pressure roll.
2. Metering of adhesive may be accomplished from front side of corrugator while running.
3. A load compensating device to relieve the Johnson joint is now in use. It may help minimize leaning and cut corrugations.
4. An electronic gadget for automatically indicating the caliper of the single or double-facer web.
5. Automatic web alignment instruments are under development.
6. A water-cooled splash apron device on corrugator starch pan which reduces adhesive gelatinization and keeps fingers cleaner. This was developed with the aid of Union Bag.

7. Oil heating of corrugator rolls. Such an installation will be completed by this Fall on a high-speed machine.
8. Automatic single-facer glue roll drop.

Mr. Scordas pointed out that the pressure roll should have independent loading front and back to help compensate for variations in moisture in the sheet.

The second speaker was Bill Ward of Hooper, who explained that his company considered the following developments new.

Hooper has undertaken a long range fundamental approach to the mechanism involved in forming and gluing of corrugated board. Some of the data presented are shown in the charts on corrugator roll crown and glue roll speeds.

**Roll Crown**

| *Lower* | *Upper* | *Crown* | *Load and journal* | *Remarks* |
|---|---|---|---|---|
| 10.900 | 8.393 | 0.008 | 800 | See 1 below |
| 10.900 | 8.393 | 0.012 | 1200 | See 2 below |
| 10.900 | 8.393 | 0.024 | 2400 | See 3 below |
| 10.810 | 10.269 | 0.024 | 4800 | See 4 below |
| 10.800 | 10.537 | 0.028 | 5600 | See 5 below |
| 12.375 | 12.250 | 0.016 | 6200 | See 6 below |
| 12.375 | 12.250 | 0.018 | 6200 | See 7 below |

1—Low board. Wrinkling in center. High and low all over.
2—Improved board height. Still low in center high and low in center.
3—Full height all over. Same high and low in center. Increases with speed.
4—Full height all over. High and low approximately equal.
5—Improved high and low. Difficulty handling crown at low speeds. Walking later found due to crown curve errors.
6—Improved top speed, no trouble with walking.
7—Increased bore and addition sump-improved steam joints.

**Relation Speeds Between Glue Rolls Single-Facer**

| *Machine No.* | *Glue diam.* | *Roll gear* | *Trans. diam.* | *Roll gear* | *Doctor diam.* | *Roll gear* | *Surface speed ratio* |
|---|---|---|---|---|---|---|---|
| 1 | 8.41 | 78 | 4.059 | 22 | ... | .. | 1.71 |
| 2 | 8.44 | 78 | 4.030 | 22 | ... | .. | 1.70 |
| 3 | 8.425 | 37 | ... | .. | 4.00 | 37 | 2.105 |
| 4 | 8.375 | 79 | 6.000 | 37 | ... | .. | 1.575 |
| 5 | 8.375 | 82 | 6.000 | 33 | ... | .. | 1.78 |
| 6 | 8.375 | 44 | ... | .. | 6.00 | 53 | 1.73 |
| 7 | 8.375 | 53 | ... | .. | 6.000 | 62 | 1.64 |

Remarks:
1—Positive—good spread thins same over 400 f.p.m.
2—Positive—good spread thins same over 400 f.p.m.
3—Positive or negative—good spread—highest speed juted 525 ft.
4—Positive—good spread thins over 300 ft.
5—Positive—thins over 350 ft.
6—Negative—very good spread. Minimum settings—no thinning.
7—Positive—very good spread. Minimum settings—no thinning, spray at roll nip.

In general, Mr. Ward felt that considerably more fundamental work should be done to bring to light some of the mysteries on the corrugating process. Only in this way can an intelligent approach be made to the problems that confront us. At Mr. Arbuthnot's request, Mr. Ward consented to present a lengthy paper in February dealing with the findings of his five-year investigation into the mechanisms of the corrugating process.

Harry Cohen presented the recent engineering advances of S & S.

1. A new diamond dust grinding process which enables holding tolerances of 0.0005 in.
2. An automatic roll tensioning device on shaftless roll stands. Installations in operation at Stone Container in Philadelphia and Hankins at Cleveland.
3. A corrugated roll with more pointed corrugations gave higher flat crush than conventional design.
4. A heavy duty cut-off is now under development.

Swift was represented by Mr. Thorn, Mr. Gebbie, and Mr. Lister. Their innovations are:

1. Hydraulic loaded and crownless pressure rolls. They believe this will help control vibration and reduce high-lows.
2. Micrometer gang adjustment for fingers and floating strippers.
3. Improving preconditioning equipment.
4. Use of oil spray has been found to increase flute height about 0.005 on C-flute and 0.008 on A-flute. With stiffer corr. mediums they use less crown.

Mr. Moreland of Greeneline outlined their new Greeneline stands and stated that Eddy Paper is installing them.

Greeneline also makes a turn-over stand for boxes coming off the press. It sells for about $5500.

JOHN FUKO, *Secretary*

# LETTERS TO THE EDITOR

## *TAPPI Statistics Conference*

To the Editor, *Tappi:*

This letter is written to express my appreciation to TAPPI for sponsoring, in the face of a deficit, the Statistical Committee's first seminar course in statistics.

Though I have been a member of TAPPI for quite a few years and enjoyed its many benefits, the value of the seminar was, without question, the best received by me from our organization.

Very much of the success of the seminar was due to the work and character of Geoffrey Beall and Charles Bicking, our instructors, and to those who had the forethought to engage them.

Congratulations on a fine job.

T. H. GEIGER, *Librarian and Statistician*
*Oxford Paper Co., Rumford, Me.*

## *Permanence of Paper*

To the Editor, *Tappi:*

We have been doing some research work on the possibility of reducing the rag content of our ordinary insurance policy and application forms from 100% rag stock to 75% or 50% rag—possibly even lower. I had written to the National Bureau of Standards for information in connection with this project, and they have advised me that there were two letters to the editor, relative to the permanence of rag and sulphite paper, in the April, 1954, issue of *Tappi* which would be of interest to us.

In addition, I wonder if you would supply me with the answers to the following:

1. Do you feel that a good grade of 50% rag paper as is manufactured today will be in good condition after 150 years?

2. Do you feel there is very little difference in the relative permanence of a 100% rag sheet of paper and a no. 1 sulphite sheet of paper, if the sulphite sheet is made from carefully prepared pulp and is very low in acidity? Do you have any figures which are the result of laboratory tests, etc., in connection with this subject?

It has come to my attention that quite a few life insurance companies have been changing the rag content of their policy and application papers from 100% rag to 75, 50, and 25% rag, because they have come to believe that 100% rag paper is not necessary to provide sufficient permanency for these documents. What is your opinion in this connection?

Any information you will be willing to furnish me regarding the durability of sulphite vs. rag papers or combination sulphite and rag papers vs. all rag paper will be very gratefully received.

JOHN P. BOXSHUS, *Forms Coordinator*
*State Mutual Life Assurance Co.*
*Worcester, Mass.*

To the Editor, *Tappi:*

There is little question but that the Bureau of Standards has shown by its experiments that when sulphite pulps are so

purified as to give them a high alpha-cellulose content, their permanence is increased. Moreover, as the per cent of alpha-cellulose in these pulps approaches that of the alpha-cellulose content of cotton, the permanence of these fibers likewise approaches that of cotton.

Therefore, papers made from sulphite pulps that have a high alpha-cellulose content can be expected to approach closely the permanence of papers made from rag, providing, of course, that they are made carefully and with a high pH and low rosin content.

However, I believe that the catch in Mr. Boxshus' questions lies in the fact that few, if any, of the 25, 50, and 75% rag papers on the market today contain, as the balance of their furnish, wood pulp having this high alpha-cellulose content. Therefore, they cannot be expected to approach the permanence of a 100% rag paper. Obviously this same difference is true to a greater degree in all-sulphite papers.

Considering the above, it is apparent that the question whether he can use safely a paper with a lower rag content depends upon the degree of permanence that he requires and the conditions under which it will be stored.

The storage conditions have been shown to be extremely important. For instance papers stored in cities where the air contains acid from coal smoke or manufacturing processes deteriorate more rapidly than those stored away from atmosphere that is not so contaminated. Moreover, high temperatures make paper deteriorate more rapidly.

Because the life of paper depends so much on the conditions under which it is stored and because the importance of such things as alpha-cellulose content, pH, and acidity were not recognized until less than 50 years ago, it is obviously impossible to make predictions as to which papers would be in good condition after 150 years in accordance with Mr. Boxshus' first question.

The one thing that we do know is that 100% rag papers carefully prepared have been known to last for hundreds of years. Also, we are confident that sulphite papers prepared from pulps not purified so as to give a high alpha-cellulose content are not as permanent as rag. Moreover, we know that he could not count on getting such high purified pulps by merely specifying the usual grades of 25, 50, or 75% rag content papers.

I am hoping the above will answer his questions. If not, I will be glad to try to answer any specific points that I have failed to cover.

K. P. Geohegan, *Vice-President*
*Howard Paper Mills, Inc.*
*Dayton, Ohio*

To the Editor, *Tappi:*

I assume that Mr. Boxshus is interested in commercial grades of "business papers."

There is no doubt that special wood pulps are now produced which have alpha-cellulose contents approaching those of good rag pulps, but to the best of my knowledge such wood pulps are not used in the manufacture of commercial grades of business papers in the rag content field. Whether such a blend would produce paper completely acceptable to the general market, I do not know.

I do not believe that it is possible to give a quantitative answer to a specific permanence question since given quantities of alpha-cellulose and acidity are not necessarily directly proportional to a given number of years of permanence and the whole problem of permanence is affected by storage and handling conditions. To my mind, making paper to meet special chemical specifications only, and making paper to meet these chemical specifications plus normal use and handling requirements are two different problems. I feel that the high alpha wood pulps seem to be acceptable in the first instance but only time and trial can definitely prove whether or not they are acceptable in the second.

The current evidence in favor of rag includes its known performance to date, as well as the fact that it gives certain strength characteristics in addition to its permanence. Since alpha pulps are a recent development in terms of the time factor of interest to Mr. Boxshus, I do not see how a practical answer based on experience can be given to his question. I believe what evidence there is has been based on laboratory work and is limited to 15 to 25-year periods. Whether the paper so made and tested can be considered commercially acceptable and to have been subjected to the handling of a "normal business life" I would not know.

F. S. Klein, *Mill Manager*
*Byron Weston Co., Dalton, Mass.*

To the Editor, *Tappi:*

The questions regarding permanence of papers, as raised by Mr. Boxshus in his letter of August 6 to the Editor of *Tappi*, cannot be answered by simple generalities. To put it in a few words, we just do not have sufficient data, and if we did, there would still be unanswered questions.

In the first place we know that carefully made all-rag papers are permanent because specimens are in existence. That is common knowledge. However, there is no such thing as a really old and carefully made wood pulp paper as the industry is too young. Therefore, it becomes a matter of opinion as to what the permanence of such sheets might be. As Mr. Boxshus will note, in the Bureau of Standards letter to the Editor of *Tappi*, their reply is quite correctly an opinion and not a statement of fact.

Secondly, the only real reason for considering wood pulp content sheets for permanent records as against all-rag papers is cost. However, when specifications for wood pulp content sheets are made comparably high, and a reliable manufacturer adds to this a great deal of care, then the price differential may be insignificant. Parenthetically, there probably are no stock papers available on the open market that are ordinarily made to such high standards.

Furthermore, even with such precautions, the life of such sheets cannot be guaranteed and the general feeling has been that the relatively small additional cost of an all-rag sheet is cheap insurance. It is inconsistent to spend thousands for protection of records against fire and theft and object to pennies for insurance against age.

Another phase of the problem is exposure. We very glibly speak of "normal" or "average" conditions but there is actually no such thing in the case of documents distributed to the public. What we really mean is that the majority of documents, such as insurance polices, are properly stored and carefully handled, and only a few are exposed to damaging conditions.

However, acid atmospheres are very common, particularly in industrial areas but not infrequently elsewhere. "Smog" is now a word well rooted in everyday language. These acid environments can be very damaging to any paper but the important point is that the glue-sized all-rag papers undoubtedly suffer the least under such conditions.

Where permanence is desired it would seem common prudence to plan for a margin of safety and not just for "average" conditions. Trouble and inconvenience from only a few cases of exposure to damaging conditions can offset any cost saving from the use of cheaper papers.

Another point which has not been given proper emphasis is the combination of strength and permanence. One of the very things that can contribute to permanence is strength because a strong sheet can lose a portion of its strength under poor storage conditions and still be serviceable. Documents, with rare exceptions, are not much good if they cannot be handled and a sheet that is not strong to begin with is not permanent in the real sense of the word. Here again the best glue-sized all-rag papers have a combination of strength and permanence that is not obtainable in other sheets.

Mr. Boxshus has read, no doubt, various comments on the significance of alpha-cellulose, low acidity, and low rosin content. In the early days of impure wood pulps a reason was sought for the lack of permanence of such wood pulp papers. Rag papers were examined for comparison and the most permanent ones were found to have a high alpha-cellulose content, a low acidity, and a low rosin content. *It has been assumed,* therefore, that wood pulp papers showing similar characteristics will approach rag papers in permanence but the highly purified wood pulps are relatively new and this assumption has not been correlated with experience as yet. There has not been time.

The matter may be summed up by saying that high-grade, glue-sized all-rag papers have long since been proved permanent. They are also strong, durable, and resistant to damaging conditions. Wood pulps have been greatly improved in recent years and the life expectancy of papers made from them has been increased—how much we cannot say with accuracy. However, to consider permanence alone and to assume "average" conditions of storage and handling is in our opinion and our experience a mistake. When the problem of permanent records is considered in all its aspects nothing but the high quality all-rag papers will answer.

Francis L. Simons
*Crane & Co., Inc.*
*Dalton, Mass.*

## *Laminating Adhesives*

To the Editor, *Tappi:*

I would appreciate it if you could help me in obtaining certain information.

My managing director has heard of a new adhesive used in the converting field for laminating two types of paper or board together. The adhesive is cold setting and requires no heat whatsoever. The impression I received was that this is used in the corrugating field or the solid container field.

H. Tanner, *Buyer*
*The Thompson & Norris Mfg. Co. Ltd.*
*Brentford, England*

To the Editor, *Tappi:*

I have just read the letter from the Thompson & Norris Mfg. Co., Ltd. in which they asked about a new adhesive which is cold setting and requires no heat.

It is very difficult to answer their question with the limited information they give. I will try to give my general impression of the adhesive field and perhaps this will cover the subject for them.

In corrugating there are two principal types of adhesives used today—sodium silicate and starch. Silicate performs best when heat is supplied to the combined paper, although it would be possible to make a satisfactory bond without heat. Starch adhesive will not work at all without heat and it is necessary not only to heat the combined paper to gelatinize the starch but also to preheat the paper going into the corrugating machine. All corrugating machines that I have ever seen in this country are equipped with hot plate sections for heating the combined board.

In solid fiber laminating the principal adhesives used are starch base products which are composed of modified starch, borax, alkali, etc. These must be cooked in water to prepare a paste and then this paste applied to the various plies making up the final product. No heat is required for the setting of the adhesive and a good many laminating machines in this country do not have any heating equipment.

While the above two adhesives comprise by far the major adhesives used in this country, they probably are not the type of product inquired about, since by no stretch of imagination, would they be considered new. Some of the new adhesives which I have seen are as follows:

1. Adhesives based upon sulphite spent liquor. (This type of product has been proposed both for corrugating and solid fiber laminating but is not used to any extent at present to my knowledge.)
2. Polyvinyl alcohol adhesives have been used for both corrugating and laminating. These do not require heat for setting. (You might write du Pont for information on this.)
3. Resin emulsion adhesives. This type of adhesive is very rapid setting and does not require heat. (National Adhesives is one of the big promoters of this type of adhesive.)
4. Soybean protein–soy flour adhesives have been used to a limited extent. (These were developed by the Glidden Co.).

Any one of the above types of new adhesives could be the one in which the Thompson & Norris Co. is interested.

J. P. Casey, *Director of Technical Service*
*A. E. Staley Mg. Co*
*Decatur, Ill.*

To the Editor, *Tappi:*

We believe that the new adhesive which the managing director of Thompson & Norris evidently has in mind is the polyvinyl resin type of which our Aquabond is an outstanding example.

Aquabond is marketed as a dry powder packed in 50-lb. bags and is prepared for use by cooking with water at recommended concentrations for specific jobs. We manufacture several types of Aquabond ranging from the very concentrated and fast setting Aquabond 3-30 to much less concentrated and somewhat slower setting Aquabond 4-55. The average price of these products is $27 per 100 lb. f.o.b. New York.

Our Aquabond adhesives are widely used for laminating solid fiberboard for the manufacture of containers. Aquabond has excellent adhesion properties and handles all grades of wet-strength paper made by the melamine or urea-formaldehyde process, which we thus far encountered. Aquabond produces a water-resistant bond which will withstand 24 hours' immersion in water without delaminating. A unique feature of Aquabond is that it attains this water resistance as soon as the bond is dry—no prolonged aging is required.

Aquabond produces excellent "mileage." Under average conditions, 1 lb. of the dry Aquabond will produce sufficient solution to handle 1000 sq. ft.

Although Aquabond is usually prepared for use by cooking with water to a temperature of 190°F. and is usually run on the machine at a temperature of 100 to 140°F., no other heating is required.

J. A. Lowander, *Adhesive Dept.*
*Stein, Hall & Co., Inc.*
*New York, N. Y.*

To the Editor, *Tappi:*

We have your inquiry with regard to the letter from the Thompson & Norris Mfg. Co., Ltd. of Middlesex, England, referring to adhesives for laminating two types of paper or board together which can be done cold. We are not sure exactly what this account is interested in but can say that in this country we use what would be called cold adhesives for several applications.

The first type of product would be a product such as our Aqua Flakes which are cold water soluble types of dextrine and are used in this country for laminating fiberboard in a number of operations. These products vary in their water absorption depending upon the particular requirements of customers.

It is also usual in this country to use resins such as our Resyn 10R3001 for some high-speed application requiring water resistance to meet Government specifications.

H. Sterne, *Sales Dept. National Adhesives Div.*
*National Starch Products, Ind.*
*New York, N. Y.*

# Tensile Strength of Paraffin Wax

*TAPPI Tentative Standard T 644 m-54**

(THIS STANDARD IS UNDER THE JURISDICTION OF THE WAX TESTING COMMITTEE)

THIS method describes a procedure for obtaining an empirical measure of the tensile strength of paraffin wax having a ductility not exceeding approximately 1/8-inch elongation at the specified temperature and relative humidity of the test, and having TAPPI melting points (TAPPI Standard T 630 m) in the range of approximately 120 to 150°F.

A set of six specimens per test sample are cast with 1/4-square inch cross-section molds of specified shape, and aged for 2 hours in a room held at 73°F. and 50% relative humidity. Tensile strength is measured by applying a load of 20 pounds per second, and is reported in pounds per square inch (p.s.i.) by converting the breaking load of a 1/4-square-inch cross-section specimen to a 1 square inch basis.

Such factors as manner and speed of testing, shape and cross section of specimen, manner of specimen preparation, conditioning of the specimen, surface condition of the specimen, and the presence of strains in the specimen, all influence the magnitude and precision of test results and must therefore be carefully controlled.

## DEFINITION

Tensile strength is the longitudinal stress (ultimate pure tensile load) required to break a representative specimen of specified shape and cross-sectional area.

## APPARATUS

1. *Testing Machine.* Any universal testing machine may be used, provided it is accurate to 1% of the minimum load to be applied, and provided that the load is applied at a constant, uniform rate. The resultant direction of the applied tensile load shall coincide with the longitudinal center line of the test specimen, and shall permit alignment to be adjusted rapidly and accurately. The machine shall be of such a design that it can be readily calibrated with standard dead weights over the entire range of loads to be applied.

2. *Specimen Molds and Cover Plates.* For casting the test specimens; constructed of half-hard brass in accordance with the requirements shown in Fig. 1. A half-hard brass cover plate 1 1/2 by 2 1/4 by 1/4 inches shall be supplied with each mold.

3. *Specimen Grips.* Universally self-aligning type, for fastening the test specimen to the testing machine. A suitable mount with universal self-aligning pivot

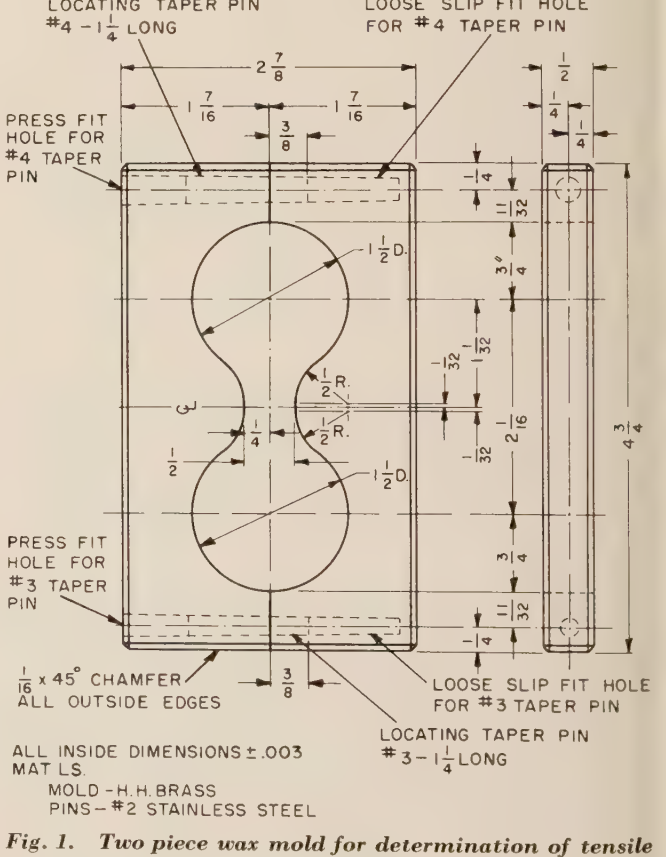

LOCATING TAPER PIN #4 - 1 1/4 LONG
LOOSE SLIP FIT HOLE FOR #4 TAPER PIN
PRESS FIT HOLE FOR #4 TAPER PIN
PRESS FIT HOLE FOR #3 TAPER PIN
1/16 x 45° CHAMFER ALL OUTSIDE EDGES
LOOSE SLIP FIT HOLE FOR #3 TAPER PIN
LOCATING TAPER PIN #3 - 1 1/4 LONG
ALL INSIDE DIMENSIONS ± .003
MAT LS.
MOLD - H.H. BRASS
PINS - #2 STAINLESS STEEL

*Fig. 1. Two piece wax mold for determination of tensile strength*

arrangement between the specimen holders and machine heads is shown in Fig. 2.

4. *Cooling Rack.* Of metal rod, constructed as shown in Fig. 3.

5. *Base Plate.* For supporting the molds in the rack, constructed of 1/8-inch No. 302 stainless steel as specified in ASTM Specifications A 167 for Corrosion-

* This method has been approved as a tentative standard by the Standards Committee. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York 17, N. Y.

Resisting Chromium-Nickel Steel Plate, Sheet, and Strip. The width of the base plate shall be equal to the length of the mold, and its length shall be equal to six times the width of the mold, plus $3\frac{1}{2}$ inches.

6. *Mold Spacers.* Of oak wood, $\frac{1}{2}$ inch square, and equal in length to the length of the mold, for uniformly separating the molds on the base plate.

7. *Water Bath.* Capable of maintaining a temperature of 73 ± 0.5°F., for conditioning the test specimens.

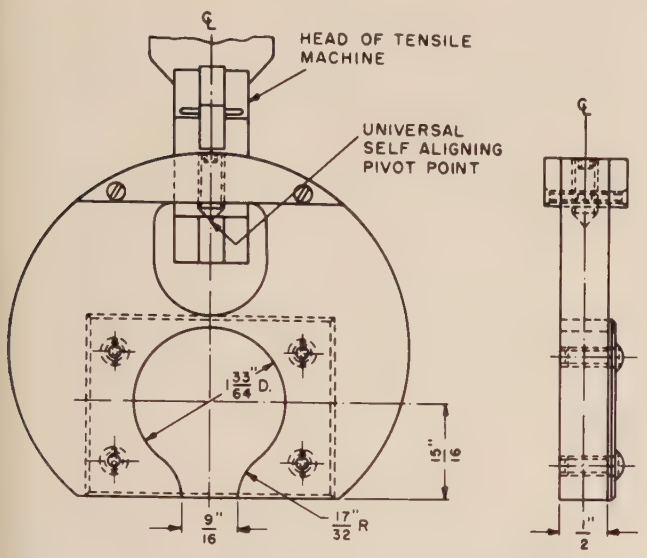



*Fig. 2. Specimen holders: two required, one upper and one lower; material aluminum 24ST*

It shall be entirely covered, and equipped with a floating grid to assure complete submergence of the test specimens. Dimensions of the bath are not critical, but, preferably, the material used in its construction should be of stainless steel.

8. *Oven.* Capable of maintaining a temperature of 212 ± 5°F.

9. *Thermometers.* For the water bath, the ASTM Precision Thermometer, having a range of 25 to 85°F., graduated in 0.2°F. intervals, and conforming to the requirements for Thermometer 63F. as prescribed in ASTM Standard Specifications E 1, shall be used. For wax stirring, the ASTM Partial Immersion Thermometer, having a range of 0 to 302°F., graduated in 2°F. intervals, and conforming to the requirements for Thermometer 1F as prescribed in ASTM Specifications E 1, shall be used.

10. *Test Room.* Maintained at a temperature of 73 ± 1°F. and a relative humidity of 50 ± 5%.

*Note:* Tests may be made for routine control or investigation purposes at other temperatures and humidities, provided the temperature does not vary more than ±1°F., nor the relative humidity more than ±5% during the entire test. Results, however, should not be considered comparable to those obtained in this test.

## CALIBRATION AND INSPECTION OF APPARATUS

### *Calibration*

Carefully check the testing machine for alignment. Calibrate it, not more than 3 months before a given test, with certified standard dead weights. Use no less than four dead-weight combinations for calibration, and select them to cover the operating range in equal increments.

### *Standardization*

In addition to calibration, the apparatus should be standardized, within 1 month of the given test, with standard waxes having tensile strengths in the normal operating range of the test. Standard samples should be prepared and retained by individual laboratories, and should be reviewed at least once a year.

*Note:* Standard samples of high- and low-tensile strength waxes are being retained at present by Section I on Strength Tests, of Technical Committee M, ASTM Committee D-2. Limited quantities of these standards are available on request to Esso Standard Oil Co., Bayonne Refinery Laboratory, Bayonne Refinery, Bayonne, N. J.

### *Mold Inspection*

Check the molds, cover plates, and base plates periodically to determine the absence of dents, nicks, and scratches which could cause points of stress concentration in the test specimen.

### *Glassware Cleaning*

Glassware should be set aside and used only for this test. It is imperative that it be cleaned properly to avoid errors due to contamination. To clean glassware, heat gently over a Bunsen burner to slightly above 212°F., and then wipe clean with a lint-free cloth.

## PREPARATION OF TEST SPECIMENS

Carefully scrape any adhering wax from the molds and wipe them clean with a lint-free cloth. Film-coat the insides of the brass molds and brass cover plates and the top of the stainless steel base plate with mold-release compound (Dow Corning Mold-Release Compound No. 7, or any other equivalent compound). Apply only a very thin film by wiping with a cloth or pad saturated with, but not containing an excess of, mold-release compound, and then wipe with a clean, dry, lint-free cloth.

Assemble the molds and wood spacers on the base plate, and the base plate on the cooling rack as shown in Fig. 3. Place the brass cover plates over the center of the molds so that the length of each plate is perpendicular to the length of the mold. Place the cooling rack and mold assembly in an area free from vibration. Place a set of prepared molds on the cooling rack for at least 1 hour before use.

Select an amount of sample sufficient to cast six specimens (approximately 200 grams). Obtain a different portion of the sample for each set of six specimens. If the sample is 1 pound or less, melt the entire amount; if greater than 1 pound, take extra precautions to obtain a representative sample in accordance with applicable quartering techniques. Transfer that portion of the wax sample to be tested to a clean glass beaker and cover with a clean watch glass; melt each 6-specimen portion in a separate beaker. Place the beaker for 2 hours in an oven regulated at 212 ± 5°F.

After 2 hours in the oven, the wax sample will be completely melted, and the temperature of the bulk of the wax will be at approximately 180°F. At this time, remove the beaker from the oven and decant all but approximately 20 ml. into a second clean glass beaker

Heat gently to 230 ± 2°F. with very mild stirring on a hot plate, using an asbestos pad. If required, filter the wax at this temperature through coarse filter paper placed in a 125-mm. diameter glass funnel with a 10-mm. stem. Discard the first 20 ml. Reheat the sample to 230°F. Use the ASTM Partial Immersion Thermometer 1F for stirring, and adjust the hot plate to heat the wax at the rate of 10 ± 2°F. per minute.

With very mild stirring, carefully adjust the sample temperature to 230 ± 2°F. and pour rapidly, filling

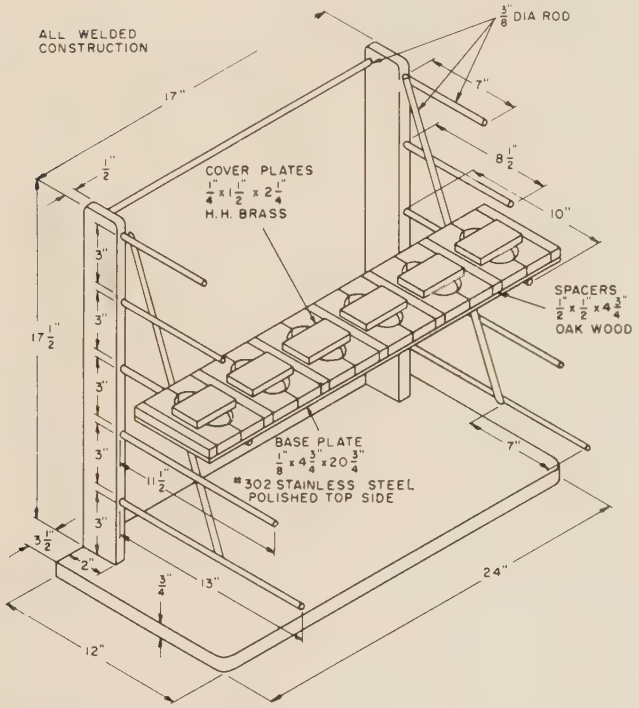



***Fig. 3. Cooling rack: base plate and mold assembly, one for each shelf***

as full as possible, without overflowing, into six previously conditioned molds placed in the cooling rack. Use the thermometer as a pouring aid. Do not disturb the cooling rack and mold assembly in any manner after the sample has been poured. Record the ambient temperature and relative humidity at the time of pouring. After 2 hours ±10 minutes, carefully remove the wax specimens from the molds and place them in the water bath, maintained at 73 ± 0.5°F. for 15 minutes, before breaking in the testing machine. Immerse the specimens completely in the water by placing them under the floating grid.

## PROCEDURE

Seat the specimen firmly and uniformly in the holder attached to the heads of the testing machine, so that the resultant direction of the applied tensile load coincides with the longitudinal center line of the test specimen. Immediately after the specimen is mounted, load the machine at a rate of 20 pounds per second for the standard $^1/_4$-square-inch cross-section mold. Record the machine load at the moment the two portions of the test specimen are separated, and the nature of the fracture. Immediately repeat the tensile measurement on the remaining five specimens for the given test. Record the average ambient temperature and relative humidity at the time the tensile measurements are made.

Calculate the mean of the results of the six tensile measurements in pounds, as read from the machine. Discard the result for any specimen whose value differs from this mean by more than 10 pounds. Record the mean of the results for the remaining specimens as the value for the sample. If results of more than two specimens are discarded from any set of six, discard all six results of that set.

## REPORT

The report shall include:

1. The mean load value for each set of test specimens, calculated for a 1-square-inch cross-section, to the nearest 1 p.s.i.

2. The predominating type of fracture, as clean or irregular; and the average distance of the fracture from the center of each of the six specimens to the nearest $^1/_8$ inch.

3. The room temperature and relative humidity at the time of pouring the test specimens and at the time the tensile measurement is made, if they differ from 73 ± 1°F. and 50 ± 5% relative humidity. (Results obtained under such conditions shall not be considered as conforming to the requirements of this method.)

## PRECISION

The following criteria should be used for judging the acceptability of the mean of results of tests on sets of four to six test specimens: Within the tensile strength range of 150 to 450 p.s.i., the means of duplicate sets of results by the same operator should be considered suspect if they differ by more than 24 p.s.i.; and the means of pairs of sets of results obtained in different laboratories should be considered suspect if they differ by more than 28 p.s.i.

## ADDITIONAL INFORMATION

This method may also be used for other types of petroleum waxes, compounded waxes, or waxes with TAPPI melting points in excess of 150°F., provided they are of the same order of ductility, and provided they do not exhibit excessive contraction on cooling the cast specimen. Certain mixtures of paraffin and microcrystalline waxes will give lower tensile strength values when using the silicone mold-release compound as prescribed in the first paragraph under Preparation of Test Specimens. While this method may be used for such mixtures, results should not necessarily be considered comparable to values obtained by this test on unmodified crystalline paraffin waxes. For waxes which exhibit excessive contraction, special casting techniques beyond the scope of this method are required.

Many purchasers and manufacturers have found the tensile strength as obtained by this method to be useful as a guide in controlling the over-all quality of paraffin wax. Because the conditions of preparing the sample and applying the load to the test specimen are not necessarily identical with service conditions, however, the tensile strength values here obtained generally cannot be assumed as indicative of the performance which could be expected in service, although some laboratories have observed a correlation in certain applications.

# Analysis of Clay

## *TAPPI Tentative Standard T 645 m-54**

**(THIS STANDARD IS UNDER THE JURISDICTION OF THE NONFIBROUS MATERIALS TESTING COMMITTEE)**

CLAY is widely used in the paper industry for a variety of purposes. It is the principal pigment component of mineral coatings on both paper and paperboard and is used in large volume as a mineral filler.

Clay is a generic term relating to a group of hydrous silicates of alumina. The species of primary interest in the paper industry is kaolin, a finely divided mineral which is capable of forming a more or less plastic mass when mixed with water.

In general, physical properties rather than chemical composition are significant as regards the suitability of a clay for use in paper. It is normally desirable that it have a good brightness and that it be free from grit. Moisture content has at least economic importance. For certain applications it is desirable to know the pH of the clay, the particle-size distribution, the viscosity when mixed with water, and the ease with which the clay can be disintegrated in water (sedimentation test).

This method covers sampling, moisture pH value, and grit. For other tests *see* T 646 m, Brightness of Clay; T 649 m, Particle-Size Distribution of Coating Clay; T 648 sm, Viscosity of Coating-Clay Slurry; and T 647 sm, Gross Particles in Coating Clay.

### SAMPLING

Clay is shipped in a variety of ways, which include bags, bulk, and slurry. Each type of shipment requires a different sampling procedure.

For bag shipments, obtain a gross sample of not less than 25 pounds. Take the samples from bags chosen at random, the number of bags so sampled being not less than 1% of those in the lot, and not less than six bags for lots of six bags or more. Blend and quarter down the gross sample to a size commensurate with the quantity needed, using procedures such as those found in TAPPI Standard T 605 m for sampling of coal.

When necessary to sample from bins or piles, or from bulk shipments before unloading, take individual samples with a trier or "thief." A device of the general type of the grain sampler recommended by the Association of Official Agricultural Chemists is usually satisfactory, but care must be taken that the openings in the sampler are large enough to give ready access to the largest lumps in the clay being sampled. Take samples at not less than six locations throughout the pile or car, using a trier of suitable dimensions to permit obtaining samples representing a cross section of the pile from top to bottom at each point sampled.

For bulk shipments, take at least eight individual samples representing the shipment. Blend and quarter down the gross sample as above.

For shipments in slurry form, a single sample is sufficient but the slurry should be well mixed before the sample is taken.

### MOISTURE

Clays contain water in two forms—free water (moisture) and chemically combined water. The first is driven off by heating at about 105°C., the second, being a part of the crystal structure, is driven off only at elevated temperatures. This test is for determination of the free-moisture content. Several methods have been proposed, including toluene distillation (*1*), oven-drying (*1*, *2*) and electrical capacitance methods. Of these, the oven-drying method is the most reliable, provided that sufficient time for complete drying of the sample is allowed.

#### *Apparatus*

1. Balance, sensitive to 10 mg.
2. Weighing dishes, aluminum or glass; or 250-ml. beakers.
3. Drying oven equipped to operate at 105 ± 3°C.
4. Desiccator.

#### *Procedure*

Weigh duplicate samples of 100 ± 0.05 grams into tared weighing dishes or beakers. Place in the drying oven and heat at 105 ± 3°C. for at least 2 hours. Cool in a desiccator and weigh. Repeat the heating for 2-hour periods until the weight is constant.

#### *Report*

Calculate the loss in weight of each sample to percentage and report the average to the nearest 0.1 as free moisture. The results of duplicate tests should agree within 0.2.

*Note:* A control procedure with a precision of about 0.4% is the toluene distillation method described in TAPPI Standard T 208 m. It is somewhat more rapid than the oven-drying method and usually is satisfactory.

### pH VALUE

The pH value of a clay-water suspension provides information which may be of importance in connection with the performance of the clay either as a filler or in a coating composition. The determination is best made electrometrically. Among other factors, the pH value obtained will be influenced by the concentration of the suspension and the length of time the suspension is allowed to stand prior to taking the pH reading.

* This method has been approved as a tentative standard by the Standards Committee. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York 17, N. Y.

*Apparatus*

1. Electrometric pH meter, glass-electrode type. A number of suitable instruments are available.
2. Balance, capable of weighing to 0.1 gram.
3. Glass-stoppered flask or cylinder, 100-ml.
4. Beaker of size suitable for containing the sample being measured on the pH meter.

*Distilled Water*

Freshly boiled distilled water is required. The water should be boiled until its volume is reduced about one third to insure removal of $CO_2$, and then rapidly cooled to room temperature.

*Procedure*

Place 20 ± 0.2 grams of the sample and 80 ml. of distilled water in the flask or cylinder. Insert the stopper and shake vigorously to suspend the clay in the water. Immediately transfer a suitable quantity of the suspension to the beaker and measure its pH with the glass-electrode instrument.

*Report*

Report the pH to the nearest 0.1 unit.

## GRIT

For this determination (*3*) all material held on a 325-mesh sieve is regarded as grit. A series of sieves is used in order to fractionate the grit according to size. Knowledge of the total quantity of grit and its size distribution is helpful in determining the suitability of clays for use both as fillers and in coating.

*Apparatus*

1. A paint shaker, or "conditioner." A typical shaker operates at 1350 shakes a minute, the shaking motion being simultaneously vertical and lateral. (A Red Devil No. 30 Shaker, manufactured by Red Devil Tools, Irvington, N. J., has been found to be satisfactory.)
2. A supply of suitable containers in which to prepare clay-water dispersions, with water-tight closures; 4-liter or 1-gallon stainless steel beakers are very satisfactory. A metal lid of appropriate size, fitted with a gasket, provides a suitable water-tight seal when lid and beaker are clamped together in the paint shaker.
3. A round 325-mesh brass sieve about 8 inches in diameter and 4 inches deep.
4. Small circular brass sieves (3-inch diameter is suitable) with 80-, 170-, and 325-mesh wire cloth, respectively, capable of being stacked together to form a water-tight column.
5. A brass top section, fitting the sieve stack and suitable for use as a water reservoir.
6. A water-leg for the sieve stack, connected to a supply of clean water.
7. A spray nozzle which delivers a thin, flat, fan-shaped spray, connected to a supply of clean water by a rubber hose and suitable fittings.
8. Balances to weigh 5 kg. and 200 grams, the latter accurate to 1 mg.

*Deflocculant Solution*

A solution of sodium hexametaphosphate containing 5 grams per 100 ml. of solution.

*Procedure*

Clean the three small sieves, dry them in an oven at 105 ± 3°C., cool, and weigh each sieve to the nearest milligram.

*Note:* It is advantageous to use a similar (slightly lighter) small sieve as a counterweight, thereby compensating to a degree for atmospheric conditions by affording a similar surface area for condensation of atmospheric moisture.

Weigh 2000 ± 10 grams of the sample into one of the tared beakers and add 20 ml. of deflocculant solution (unnecessary if the clay is in "predispersed" form) and approximately 3 liters of clean tap water. Place the lid on the beaker and seal by clamping both together in the paint shaker. Start the shaker and operate for 5 minutes. Inspect the dispersion visually and allow an additional shaking period if undispersed lumps are evident. Shaking for 5 minutes is usually adequate and avoids disintegration of material which would be likely to remain undispersed in usual mill makedown procedure.

Remove the beaker, loosening the clamp slowly to permit dissipation of pressure without loss of sample by spraying from the edge of the closure, and wash all clay from the under side of the lid into the beaker. Place the 8-inch 325-mesh sieve on a tripod support in the sink and pour the slurry through the sieve, using a high-velocity stream of water (obtained by pinching the tip of a wash hose between the fingers) to prevent clogging of the sieve. Discontinue the high-velocity stream as soon as the slurry has passed completely through the sieve. Then thoroughly rinse and drain all traces of clay or grit from the beaker into the sieve. Use the fan-shaped spray from the nozzle to "brush" material remaining on the sieve until no further passage of material through the sieve is evident.

Next assemble the stack of weighed 3-inch sieves with the 80-mesh sieve on top, then the 170-mesh, and the 325-mesh sieve on the bottom. Place the sieve stack, including the top section (reservoir) on the water leg. Admit water to the water leg to expel air and prevent air-locks between the sieves, adjusting the flow to maintain a water level about 2 inches above the 80-mesh sieve.

Transfer the residue in the 8-inch sieve quantitatively to the sieve stack. Use a slow stream from the wash hose to flush the surface of the 8-inch sieve, controlling the rate of transfer so as to avoid overflow of the reservoir. Pinch the tip of the wash hose gently to stir the grit with the stream, thus breaking up any clumping of the residue. Then reduce the flow of water, allowing the level to fall below the 80-mesh sieve, and wash down the inside of the top section to make certain all residue is in the sieve itself.

Remove the top section and the 80-mesh sieve from the assembly and refit the top section to the 170-mesh sieve. Raise the water level above the 170-mesh sieve and repeat the stirring and subsequent steps as with the 80-mesh sieve, finally washing down the inside of the top section as before. Remove the top section and the 170-mesh sieve, refit the top section to the 325-mesh sieve, and repeat the gentle stirring and wash-down steps, finally removing the 325-mesh sieve containing the finest fraction of the grit.

Dry all sieves, with their contents, in the oven and weigh to the nearest milligram.

***Report***

Report the sieve residue (grit content) for each sieve size as a percentage of the original weight of the sample, expressed to three significant figures.

*Notes:* (1) Visual or microscopic examination of the grit reporting its apparent composition (cinders, overdried agglomerates, sand, mica, etc.) is recommended as an aid in identifying sources of grit and their elimination.

(2) Results obtained by close adherence to the above procedure have been found to be definitely more reproducible than grit determinations based upon brushing the sample through a sieve with a camel's hair or other soft brush. In addition, the relatively large specimen size affords added assurance that the value obtained will be representative of the lot being tested.


**LITERATURE CITED**

1. Lester, Ivan L., and Lyons, S. C., "Testing Clays for Paper Machine and Brush Coating," *Tech. Assoc. Papers* 30: 251–257 (1947).
2. Huber Corp., J. M., "Kaolin Clays and Their Industrial Uses," p. 42, New York, N. Y., 1949.
3. Miles, F. K., "Determination of Sieve Residue of Clay," unpublished report.


# Brightness of Clay

## *TAPPI Tentative Standard T 646 m-54**

**(THIS STANDARD IS UNDER THE JURISDICTION OF THE NONFIBROUS MATERIALS TESTING COMMITTEE)**

In this method the term "brightness" is used in the same sense as in TAPPI Standard T 452 m, Brightness of Paper. The test does not give a complete description of *color*, which can only be obtained by a spectrophotometric analysis. Nevertheless, it is a convenient means of indicating the color of commercially white materials.

An essential requirement of this test is uniformity of sample preparation, including drying, pulverizing, and preparing a test plaque. The degree of comminution exerts an appreciable influence on the results of a brightness test on clay (*1, 2, 3*). In order to achieve uniform pulverization, the use of a hammer-mill pulverizer is much preferable to grinding the specimen with a mortar and pestle or similar methods.

Drying must be sufficient to reduce free moisture to about 1.5 to 2.0%. However, care must be taken to avoid prolonged drying or subjecting the specimen to excessively high temperatures (*1, 2*).

The test plaque is formed by compacting the specimen in a metal cylinder, applying pressure with a plunger. The degree of compaction affects the brightness result and must be maintained at uniform values (*1, 2, 3*).

### APPARATUS

1. *Reflectance Meter*, so designed and constructed as to permit operation in compliance with T 452 m.
2. *Pulverizer*, hammer-mill type. Examples of satisfactory pulverizers are the Raymond laboratory pulverizer, Mikro Bantam Pulverizer, and Mikro Samplmill. The pulverizer should be equipped with a screen such as the Raymond MS-104 or Mikro 039RD.
3. *Plate Glass*, 5 by 5 by $^3/_8$ inches, with a smooth, unscratched surface.
4. *Cylinder*, of brass or stainless steel, at least 1 inch long, with inside diameter slightly larger than the sample aperture on the reflectance meter, and with ends cut and faced smooth, vertical and square to the longer axis. The area of the cross section in square inches should be calculated from the inside diameter and recorded.
5. *Plunger*, of wood or metal, with flat, smooth end, of such diameter as to slip easily into the above cylinder and about 6 inches long.
6. *Drying Dishes.* Shallow, easily cleaned containers 8 to 10 inches in diameter, made of corrosion-resistant material.
7. *Drying Oven*, thermostatically controlled at 105 ± 3°C.
8. *Platform Scale*, 100-pound capacity, bathroom type, so constructed that dial can be read from above.

### TEST SPECIMEN

The test specimen shall consist of about 100 grams of air-dry clay (or the equivalent amount of clay slurry) obtained according to the sampling procedure described in TAPPI Standard T 645 m.

### PROCEDURE

Spread out about 100 grams of sample in a drying dish and dry for 1 hour at 105 ± 3°C. Take care to locate the container in the oven in a position where it does not receive sufficient radiant heat to heat the sample above 110°C. Pass the dried sample once through the pulverizer, collecting the pulverized material in a glass jar or paper bag.

Place the glass plate on the platform scale and center the cylinder upon it. Pour the pulverized sample into the cylinder to a depth of about $^1/_2$ inch and then insert the plunger, being careful to let it settle slowly to avoid blowing finely pulverized clay from the cylinder. Depress the plunger holding both plunger and cylinder to prevent tipping or sliding, until the scale shows a weight equivalent to 30 p.s.i. pressure on the area of the test plaque. Release the pressure, withdraw the plunger, and remove the cylinder containing the compacted plaque from the glass plate. Do not rotate the cylinder, as this will "polish" the surface of the plaque and lead to erroneous results.

Inspect the surface of the plaque. If it is broken, irregular, rough, or contaminated, discard it and make a new one. The surface must not be touched or marred by contact. It is convenient to set the cylinder vertically in inverted position until ready to place it on the reflectance instrument.

Carefully place the cylinder containing the plaque on the instrument and measure the brightness with ref-

* This method has been approved as a tentative standard by the Nonfibrous Materials Testing Committee. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York 17, N. Y.

erence to a calibrated working standard in accordance with operating instructions supplied with the instrument.

It is recommended that at least three plaques be prepared from each sample and that three readings be made on each plaque.

### REPORT

Report the average percentage brightness of the sample (based on magnesium oxide = 100.0), to one decimal place, together with the maximum and minimum readings.

### LITERATURE CITED


1. Lester, Ivan L., and Lyons, S. C., "Testing Clays for Paper Machine and Brush Coating," *Tech. Assoc. Papers* 30: 251–257 (1947).
2. Millman, N., "Brightness determination of paper clays," *Paper Trade J.* 127, No. 13: 43–55 (Sept. 23, 1948).
3. Edgar Brothers Co., "Degree of Precision to Be Expected in Brightness Testing," unpublished study (1949).


# Gross Particles in Coating Clay

## *TAPPI Suggested Method T 647 sm-54**

**(THIS SUGGESTED METHOD IS UNDER THE JURISDICTION OF THE NONFIBROUS MATERIALS TESTING COMMITTEE)**

In addition to contaminants such as fragments of accessory minerals, agglomerates of clay particles which are not readily disintegrated by normal coating-mill make-down procedures may adversely affect the quality of coating clays even though their size is such that they pass through the finest testing sieves. This test furnishes an index to the amount of such agglomerates present. An excessive content of agglomerates which resist the dispersive action of color-room mixing equipment is likely to cause coating-mill troubles and to produce unsatisfactory coated paper.

The test consists of applying to a fluid, deflocculated clay slurry a controlled amount of work sufficient only for the disintegration of clays which are not agglomerated, segregating the gross particles by decantation, and determining by rapid settling their apparent volume.

Agglomeration here refers to cementation of particles, as by overdrying, rather than to loose aggregates such as those resulting from flocculation. The test is primarily applicable to coating clays from which naturally occurring gross particles have been eliminated by the fractionation procedures employed in their preparation.

### APPARATUS

1. Pulverizer, hammer-mill type. Examples of satisfactory pulverizers are the Raymond laboratory pulverizer, Mikro Bantam Pulverizer, and Mikro Sampl-mill. The pulverizer should be equipped with a screen such as the Raymond MS-104 or Mikro 039 RD.

2. Constant-speed power mixer (1600 r.p.m.), such as the Model L Lightnin' mixer equipped with 2-inch three-blade impeller.

3. Low-form Pyrex beaker, 1-liter, marked at depths of 1 inch and 5 inches above the bottom.

4. Means for decanting slip from beaker. Preferably an aspirator and receiving flask connected by a rubber tube to a glass tube with its open end slightly constricted, the glass tube being so bent that the open end is level with the 1-inch mark on the beaker when the bend rests on the rim of the beaker.

5 Long-form 100-ml. centrifuge tube (ASTM D-96) and suitable stand.

6. Usual laboratory equipment, such as balance to weigh 250 grams, accurate to 1 gram; graduated cylinders; stopwatch or other timer; sample containers; and 1-quart Mason jars, or similar vessels.

### REAGENTS

*A. Tetrasodium pyrophosphate* (TSPP), 5% solution.

*B. Phenolphthalein indicator*, 1% solution in 50% alcohol.

### TEST SPECIMEN

The test specimen shall consist of 250 grams of clay, obtained according to the sampling procedure described in TAPPI Standard T 645 m.

### PROCEDURE

Place 500 ml. of tap water in a 1-quart Mason jar and add 30 ml. of TSPP solution. Weigh, to the nearest gram, 250 grams of pulverized clay and add to the jar. (Pulverized clays may be weighed directly as received. Put clay in lump or extruded form once through the laboratory pulverizer before weighing.) Immediately place the jar on the mixer, adjusting it so that the clearance between the highest point of the jar bottom and the impeller is $^{5}/_{8}$ to $^{11}/_{16}$ inch. Stir the slurry at full speed for 10 minutes.

*Note:* It is important that at this stage the slip be fully deflocculated. The quantity of deflocculant (TSPP) specified is suitable for normal coating clays of domestic origin. However, for some clays, the dosage may be either inadequate or excessive. The state of deflocculation may be estimated by the following procedure: Blow vertically, with pursed lips, on the slip with sufficient force to create a depression 1 to 2 mm. Deep at the center of impingement. When blowing is stopped there should appear on the surface an irregular, brownish, "oily" ring, indicative of streaming double refraction. With fully deflocculated suspensions the ring will collapse and disappear within 2 seconds. Slower collapse, or failure of the ring to appear, indicates incomplete deflocculation, which may be caused either by too much or by too little TSPP. In such a case add 5 to 10 ml. of TSPP solution, stir well, and re-examine. If no improvement results from one or two such additions start at the beginning of the procedure with a fresh test specimen, using a smaller quantity of TSPP solution.

* This method is suggested by the Standards Committee as being the most suitable to date. It is *not*, however, a TAPPI Standard. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York, 17, N. Y

Pour the slurry into the 1-liter beaker. Rinse the jar with small portions of fresh water, allowing the rinsings to run into the beaker, and then fill the beaker to the 5-inch level. Allow the resulting slurry to settle undisturbed for 10 minutes.

Insert the decanting tube slowly and, with its open end 1 inch above the bottom of the beaker, decant down to this depth, avoiding such rapid flow as to disturb the sediment. Refill the beaker with fresh water to the 5-inch mark, using initially a strong flow of water directly from the tap to produce turbulence for a few seconds. (This is to separate from the agglomerates as much as possible of the loose, dispersed particles.) Allow the diluted slip to settle undisturbed for 5 minutes. Repeat the decantation, gently tilting the beaker and manipulating the decanting tube to remove as much as possible of the suspended fine particles without disturbing the gross sediment.

Transfer the sediment from the beaker to the centrifuge tube, flushing the residue into the tube with one or two small portions of water from the tap. Fill the tube to the 75-ml. mark with tap water and add TSPP solution to the 100-ml. mark. Add a few drops of phenolphthalein solution to produce a medium rose color and facilitate the reading of the volume of sediment. Cover the mouth of the tube with the thumb and shake the tube vigorously for a few seconds. Then immediately place the tube in its stand and simultaneously start the stopwatch.

The settled particles will appear white in the tip of the tube. Read the apparent volume of sediment 1 minute and again 10 minutes after settling begins.

### REPORT

Report the apparent volume of sediment in milliliters, to the nearest 0.01 ml., for the respective settling times.

### ADDITIONAL INFORMATION

The significance of the readings at 1 and 10 minutes lies in the fact that particles of clay, whether undisintegrated or discrete, tend to settle at a rate directly as the square of their diameters. For this reason, readings are taken at two time points in order to determine whether the majority of the particles are large or whether there is a large amount of small particles. The large particles are caught in the 1-minute reading; the small particles tend to fall out and will be included in the volume at the 10-minute reading.

There are times when a few large particles would be unobjectionable, while at the same time a large number of small particles would make a clay totally unacceptable. For example, a few large particles might cause a few holes in the coating of a sheet; on the other hand, the presence of a large amount of small particles, while possibly unnoticeable in the sheet, may cause severe dusting on the calenders and deterioration of the coating.

# Viscosity of Coating-Clay Slurry

***TAPPI Suggested Method T 648 sm-54****

**(THIS SUGGESTED METHOD IS UNDER THE JURISDICTION OF THE NONFIBROUS MATERIALS TESTING COMMITTEE)**

Flow properties of coating clays are of importance to the paper coater in that they affect the functioning of the coating operation. While it would be ideal to determine the viscosity of a complete coating mixture containing the clay in question, sufficiently standardized adhesives and methods for preparing them in solution form have not been developed to the point where the average laboratory in the paper or clay industries could reliably use such a method for general purposes. Consequently, it seems preferable to measure the flow properties of a completely dispersed aqueous suspension of the clay.

This test includes the accurate preparation of an aqueous suspension containing 71% solids, with introduction of the dispersing chemical in increments to obtain the optimum dosage (minimum viscosity) and the determination of that viscosity. It is important to obtain with precision this high solids content of the clay slurry, since differences in clays which would not be apparent at lower solids contents are magnified in this range. It is also important to accurately measure the dosage of dispersing reagent added, since small differences from the optimum may cause large differences in the resulting viscosity.

Not all clays will form slurries of such high solids content sufficiently fluid to permit viscosity readings with the usual instruments. The test as written is thus substantially limited in its applicability to the type of coating clays suitable for high-solids coating, usually domestic in origin and processed so as to have particle-size distributions imparting desirable high-solids flow characteristics. A similar test procedure using lower solids content is informative in the case of clays not suited to testing at 71% solids, but in each case the solids content should be maintained at as high a level as possible to accentuate differences between the clays in question.

Several types of viscometers are in use in the industry, the Brookfield, Stormer, and MacMichael being the principal types. However, clay slurries usually are not true viscous systems, and display non-Newtonian types of flow. It is, therefore, necessary to carefully control the rate of shear in order to accurately and reproducibly measure flow properties. The Brookfield is particularly suitable for this purpose and has been arbitrarily selected as the standard instrument.

* This method is suggested by the Standards Committee as being the most suitable to date. It is not, however, a TAPPI Standard. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York 17, N. Y.

## APPARATUS

1. Brookfield Special four-speed viscometer, Modified Model RVF, operating at 10, 20, 50, and 100 r.p.m., and equipped with a No. 3 spindle.

2. Heavy-duty high-speed mixer with variable speed. Mixers with optimum performance characteristics plus construction rugged enough to withstand fairly constant usage are not easily located. A mixer such as the Waring Blendor, Hamilton-Beach No. 30 drink mixer, or the type of mixer called for in ASTM procedure D-422 and provided with a variable voltage transformer for speed variation is satisfactory but too frequent use may result in overheating. A thoroughly satisfactory mixer can be built with a $^1/_4$-hp., 10,000-r.p.m., series-wound motor equipped with a four-blade impeller of the malted-milk mixer type, about $^7/_8$ inch in diameter.

3. Pycnometer. An aluminum pycnometer such as Fisher No. 3-247 is satisfactory.

4. Usual laboratory equipment, including a balance with a capacity of 2000 grams; analytical balance sensitive to 1 mg.; 200-mesh sieve; thermometer graduated from 0 to 100°C.; beakers, pipets, and 25-ml. buret.

## REAGENT

*Dispersant solution*, 0.5 gram per ml. While a number of dispersing agents are suitable in that they produce deflocculated suspensions, the test procedure requires a concentrated solution in order to avoid undue dilution as increments of dispersant are added. Sodium hexametaphosphate and sodium tetraphosphate are suitable.

## TEST SPECIMEN

The test specimen shall consist of the equivalent of 500 grams of moisture-free clay, obtained according to the sampling procedure described in TAPPI Standard T 645 m.

## PROCEDURE

Determine the moisture content of the sample; then weigh out the equivalent of 500 grams of dry clay.

Place 190 ml. of distilled water in the mixer cup; but if the moisture content of the clay is greater than 2%, decrease the quantity of water correspondingly. Pipet into the cup 2.0 ml. of dispersant solution, equivalent to 0.2% of dispersant on the dry weight of the clay. Then place the cup on the mixer and start the motor at low speed. Add the clay in small increments, allowing it to mix in thoroughly between additions. When all of the clay has been added, agitate the mixture at high speed (approximately 7000 r.p.m.) for 30 minutes; then remove the cup from the mixer and screen the slurry through the 200-mesh sieve.

Determine the specific gravity of the slurry with the pycnometer. Weigh out 310 grams of the slurry and add from a buret the correct additional quantity of water (*see* Table I) to adjust the solids content to 71%. Mix at low speed for a few minutes to distribute the dilution water throughout the slurry, and then adjust the temperature to 26 ± 1°C.

Measure the viscosity on the Brookfield viscometer at 10 and 100 r.p.m., respectively, using the No. 3 spindle, and record the viscosity readings. Pipet into this slurry 0.22 ml. of dispersant solution, equivalent to 0.05% on the dry weight of the clay, thereby increasing the dispersant dosage to 0.25%. Again measure and record the viscosity.

Repeat the addition of 0.22-ml. increments of dispersant solution and viscosity measurements after each addition until an increase in viscosity is noted, to establish the dispersant dosage for minimum viscosity. Viscosity readings should be made at at least five dosages, for example, 0.20, 0.25, 0.30, 0.35, and 0.40%, respectively.

**Table I. Additional Water to Adjust 310 Grams of Clay Slurry to 71% Solids**

| *Specific gravity of slurry* | *Solids, %* | *Water to be added, ml.* |
|---|---|---|
| 1.770 | 71.00 | 0.0 |
| 1.771 | 71.05 | 0.2 |
| 1.772 | 71.10 | 0.5 |
| 1.773 | 71.15 | 0.7 |
| 1.774 | 71.20 | 0.9 |
| 1.775 | 71.25 | 1.1 |
| 1.776 | 71.30 | 1.3 |
| 1.777 | 71.35 | 1.6 |
| 1.778 | 71.40 | 1.8 |
| 1.779 | 71.45 | 2.0 |
| 1.780 | 71.50 | 2.2 |
| 1.781 | 71.55 | 2.5 |
| 1.782 | 71.60 | 2.7 |
| 1.783 | 71.65 | 2.9 |
| 1.784 | 71.70 | 3.1 |
| 1.785 | 71.75 | 3.3 |
| 1.786 | 71.80 | 3.6 |
| 1.787 | 71.85 | 3.8 |
| 1.788 | 71.90 | 4.0 |
| 1.789 | 71.95 | 4.2 |
| 1.790 | 72.00 | 4.4 |

## REPORT

Report the minimum viscosity at 10 and 100 r.p.m., and also the percentage dispersant dosage on the dry weight of the clay to obtain this minimum viscosity. The report shall also state that the viscosity was determined according to TAPPI method T 648 sm.


**40th Annual Meeting**

**of the**

**Technical Association**

**of the**

**Pulp and Paper Industry**

**Hotel Commodore**

**New York, N. Y.**

**February 21–24, 1955**

# LOCAL SECTION ACTIVITIES

*Reports of Meetings, Personnel, and Events*

## *Empire State*

The annual meeting of the Empire State Section of the Technical Association of the Pulp and Paper Industry was held at the Whiteface Inn at Lake Placid, N. Y., June 10–12, 1954. This is the meeting in which all districts of the section participate.

At the business meeting the officers for the 1954–1955 season were announced: Chairman, F. G. Sommerville, Armstrong Cork Co., Inc.; First Vice-Chairman, W. R. Willets, Titanium Pigment Corp.; Second Vice-Chairman, G. K. Storin, Niagara Alkali Co.; Third Vice-Chairman, R. H. Wiles, International Paper Co.; and Secretary-Treasurer, R. N. Prince, Eastern Chemicals, Inc.

Two papers were presented in competition for the Empire State TAPPI Award, "Pulp and Paper Mill Industrial Waste Surveys," by Gorden E. Rabeler and "The Effect of Adhesives Used on the Rheological Properties of Several Coating Colors," by Miss Paulene Chu, both of the International Paper Co. Mr. Rabeler's paper was published in the June 1954 issue of *Tappi*, pages 144A–146A. Miss Chu's paper is to appear at a later date.

The program for Friday morning, under the chairmanship of J. D. Parmele included papers by G. L. Booth, Dilts Machine Div., The Black Clawson Co., "The Application of Polyethylene and Polyethylene-Wax Blends by Extrusion and Roller Coater Methods"; J. B. Sisson, St. Regis Paper Co., "The Modification of Plastic Films for Specific Packaging Needs"; S. T. Orton, Jr., Orton Corp., "The Rotobelt," Hugh Holland, Monsanto Chemical Co., "Foam Measurement"; and B. W. Weiner, E. I. du Pont de Nemours & Co., Inc., "New Chemical Products of Interest to the Paper Industry."

In the afternoon there was a panel discussion on stream improvement. Introduced by Mr. Parmele, R. M. Drummond of International Paper Co., and V. P. Edwardes, consultant, presented brief statements covering general aspects of mill control and chemical recovery. Then W. H. Larken of the New York State Department of Health outlined the activities of the State's water control program. Finally, H. W. Gehm of the National Council for Stream Improvement presented a recorded discussion prepared by the Council on the sources of water supply. This was followed by questions from the audience.

Saturday morning S. J. Young, Crown Zellerbach Corp. was chairman. The first paper was "Odor Testing in Paperboard," by R. T. Russell, Syracuse University; followed by "Applications of the Hurletron Automatic Caliper and Basis Weight Control to Paper and Paperboard Machines," by C. M. Noble, Electric Eye Equipment Co.; "Developments in Flotation Deinking of Waste Paper," by J. W. Jelks, J. W. Jelks Co.; and "New Chemical Products of Interest to the Paper Industry," by Warren Kumblad, Socony-Vacuum Oil Co. A showing of the movie "The Ancient Art of Chinese Papermaking" by E. D. Jones & Sons completed the technical program.

The papers available for publication follow this report.

The social program started with a "get acquainted" party Thursday evening. Dances were held Friday and Saturday nights, with a costume competition Friday and awarding of the prizes, Saturday. The ladies enjoyed sessions of bridge and canasta and, for those who wished, trips to Whiteface Mountain and a boat trip on the lake. Saturday afternoon was devoted to golf and trap shooting.

The committee members for this meeting included: General Chairman, D. C. Mather, Carthage Paper Makers, Inc.; Technical Meetings—J. D. Parmele, St. Regis Paper Co., and S. J. Young, Crown Zellerbach Corp.; Reservations and Finance—G. B. Martin, F. C. Huyck & Sons, and R. N. Prince, Eastern Chemical, Inc.; Publicity—R. S. LaRue, Socony-Vacuum Oil Co.; Social Events—J. J. Lynch, Corn Products Sales Co.; Men's Golf—M. D. Meyers, Socony-Vacuum Oil Co.; Ladies Events—Mrs. J. J. Lynch and Mrs. R. N. Prince; Trap-shooting—H. D. Cook, Sweet Bros. Paper Co.; Prizes—Mr. & Mrs. Paul Anderson, St. Regis Paper Co., and Toastmaster, Homer Rice, E. I. du Pont de Nemours & Co., Inc.

### The Application of Polyethylene and Polyethylene-Wax Blends by Extrusion and Roller Coating Methods

*George L. Booth*

THE subject matter to be covered in this paper is, in general, familiar to most converters today. There are, however, new coating developments under way in which polyethylene is playing a very important role. To meet this challenge and to open new vistas of conversion methods, products, and economies, it is necessary to develop machinery that will handle these coatings satisfactorily. This presentation will cover primarily coating machinery under develop-

GEORGE L. BOOTH, Research Engineer, Dilts Machine Works Div., The Black-Clawson Co., Fulton, N. Y.

*Committee members for the Empire State Section Annual Meeting. Seated: Mrs. J. J. Lynch, Mrs. G. B. Martin, Mrs. R. N. Prince, and Mrs. L. Georgevits. Standing: R. S. LaRue, Socony-Vacuum Oil Co.; D. C. Mather, Carthage Paper Makers, Inc.; G. B. Martin, F. C. Huyck & Sons; R. N. Prince, Eastern Chemicals, Inc.; J. D. Parmele, St. Regis Paper Co.; M. D. Myers, Socony-Vacuum Oil Co.; J. H. Treadwell, Treadwell Supply Co.; J. J. Lynch, Corn Products Sales Co.; and P. L. Haggerty, Geo. La Monte & Son*

ment and in use with polyethylene and polyethylene-wax blends. To a lesser degree extrusion lamination will be discussed for these new coatings and polyethylene in order to obtain a better perspective of this dynamic subject.

The paper will be divided into three general categories: (1) roller coating methods used in handling polyethylene and polyethylene blends, (2) extrusion lamination techniques used in handling polyethylene and polyethylene blends, and (3) a comparison of these two methods showing advantages and disadvantages of each including the way in which the two methods complement each other.

Before getting into the method of coating, perhaps it would be well to show the viscosity-temperature relationship of some polyethylene resins as well as the way in which paraffin wax will alter the viscosity. Figure 1 shows the effect of molecular weight on the viscosity-temperature relationship. Note the extremely wide range of viscosity between paraffin and the polyethylenes. Figure 2 shows the effect of a paraffin additive to the resin (*1*). Note the almost linear relationship of resin blend to viscosity, and the profound difference in viscosity that addition of paraffin makes.

## ROLL COATING

There have been many different ways proposed for low percentage polyethylene-wax coating. The most common in use today is the conventional wet waxing machine shown in Fig. 3 and very adequately described recently by Haskell and Eliker (*2*). The trend is toward higher poly addition to reduce blocking, give a better heat seal, and low temperature flexibility, especially for frozen food wrap and specialty items. These machines, however, are limited in the viscosity that can be handled. A normal machine operating at 1000 f.p.m. must be reduced in speed to about 700 to 900 f.p.m. with a 3 to 5% poly addition because of the increase in viscosity. There are, however, new low molecular weight polys on the market that may soon increase the percentage of poly that can be added (*3, 4, 5*).

Other waxers such as the wire-wound bar can handle essentially the same blends as squeeze roll waxers although at lower speeds (*6*).

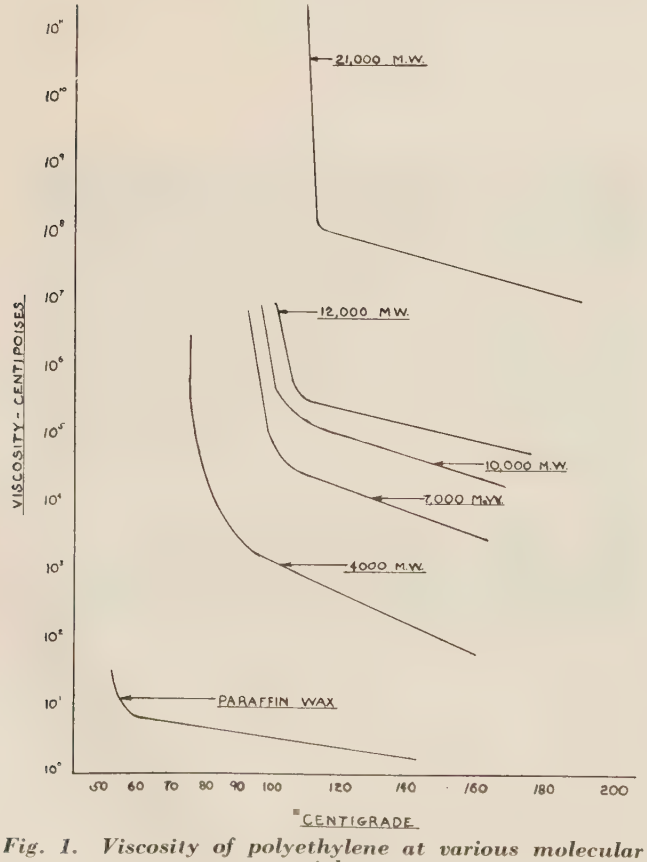



*Fig. 1. Viscosity of polyethylene at various molecular weights*

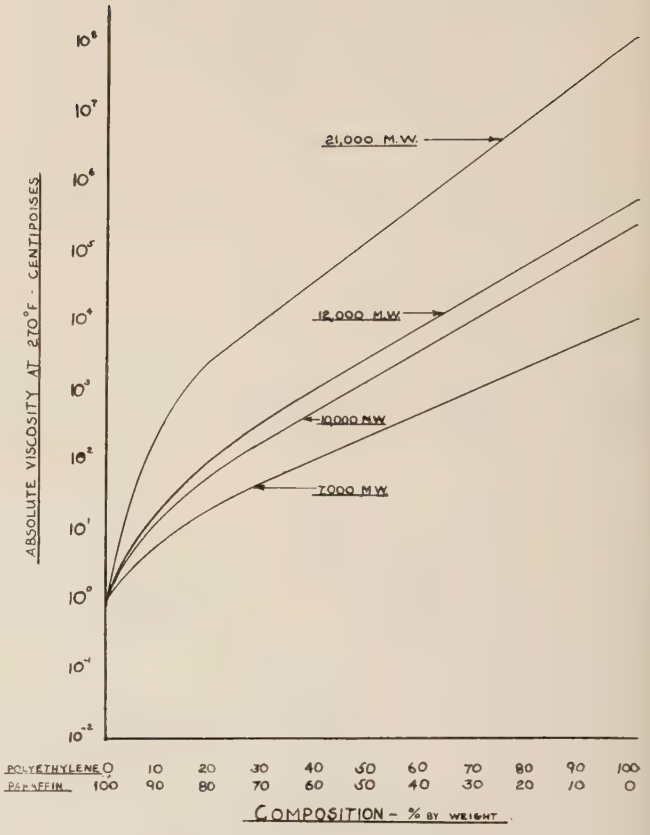



*Fig. 2. Viscosity of polyethylene-wax blends*

The development of gloss in a surface coating made on waxing machines is usually performed by passing the web through a bath of cold water if waxed on both sides, or by passing the web over a series of highly polished chill rolls to cool the coating fast enough to develop the maximum gloss.

### *Methods*

One proposed method is that shown in Fig. 4 (*7*). Originally, this method was developed for casting of unsupported molecularly oriented films from hot melts in the viscosity range of polyethylene having a molecular weight around 20,000. With slight modification, this method would be adaptable to coating a web as shown.

There are apparent disadvantages in this method as might be noted. First, is the caliper control of the coating. It would be difficult to achieve a completely pinhole-free coating under $1^1/_2$ mils and probably it would be nearer $2^1/_2$ mils. Since the web must pass through the metering device any variations in web caliper would show up in an inverse manner in the coated film. Secondly, high speeds would not be obtainable. Third, because of the difficulty in keeping heated rolls accurate, the caliper variation would be relatively great. Also, there would be a limit in viscosity below which the

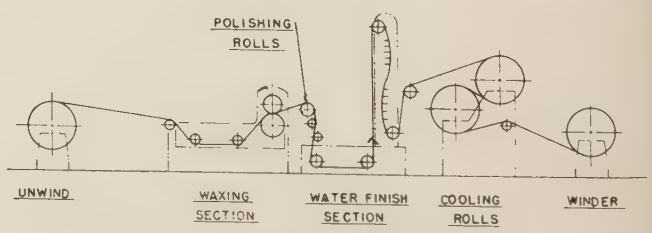



*Fig. 3. Wet wax*

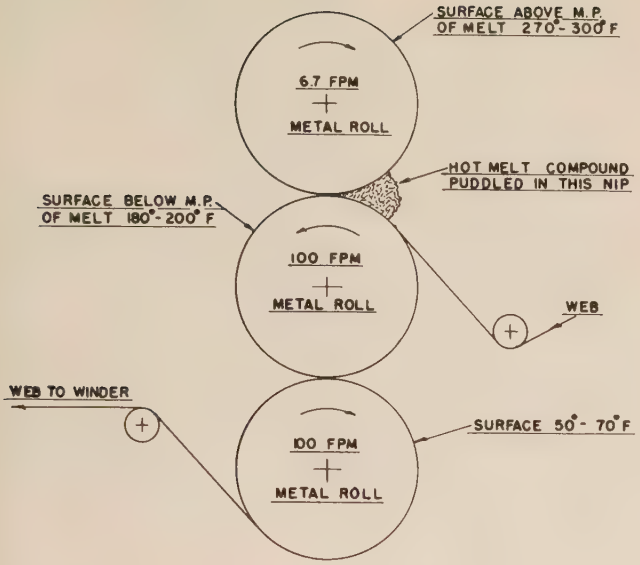



*Fig. 4. W. M. Bright, pat. 2,631,954*

method would be difficult to use since the coating solution must stay in the nip as shown.

Another method is shown in Fig. 5 (*8*). Usually, the doctor roll is run from 5 to 15% of the lineal speed of the web in the same direction. Note that the coated surface is contacted by the chill roll to give the desired gloss and surface characteristics.

The method has all the advantages of simplicity, but would be limited in speed and width. Again, since the web has to pass through the metering device, the coating would not be precisely controlled. Any variation in paper caliper would show up in an inverse manner in the coated film. With the small doctor roll, the ever present problem of roll crown and heat distortion would tend to give a nonuniform coating both down the web and across the web. Further, a limitation in viscosity would be present with this machine. If too thin, it would be difficult to maintain the puddle, and if too thick, a ridging of coating on the coated sheet would result unless further smoothing were undertaken.

A further method that might have some application, however specialized, is shown in Fig. 6 (*9*). Essentially, the film would be cast on an endless belt, probably metal, smoothed out by heat, and then transferred to the web. After cooling, the coated sheet would then be stripped from the belt. This method was originally developed for solvent coatings of relatively high viscosity, but it would appear that it might be used for hot melt work with some application.

In addition to the method of roll coating just explained, a gravure coater would be applicable for some poly-wax applications. The disadvantage of such a method would be the limitation to only one coating weight unless a supply of different rolls were on hand and the application in a relatively low viscosity range. Further, it would be necessary under most conditions to follow the coating application with a smoothing bar to achieve a smooth and continuous film. This method is used to a limited extent in waxing operations and could probably be applied to low molecular weight polyethylenes. The temperature of operation would also be limited by the heat to which a rubber roll was subjected.

The gravure method does present some very strong advantages. First, a uniform coating weight is always applied, and second, heat distortion is not a serious problem since the coating is dependent on the pattern engraved in the roll.

Van Guelpen in a recent patent explained an interesting coating machine for handling rather viscous hot melts. This particular machine is shown in Fig. 7 (*10*). The principal advantage claimed in this machine was no lodging of foreign matter in a nip or die to give voids or scratch marks in the coated sheet. Since no work has been done in our lab with such a machine, very little can be said about the method.

Referring to Fig. 7, the operation of the machine is as follows. The coating is fed by suitable means into either or both ends of the barrel and forced through the perforated slot into the outer chamber and then into the die. As shown in the plan view, the perforated slit is made in a slight arc with holes of diminishing size toward the ends. By rotation of the inner barrel, the width may be accurately deckled to suit the width of the web being run. Then, by rotating the assembly on its trunions to vary the angle at which the web passes over the smoothing bar or leveling portion of the head a greater or lesser coating weight is deposited. Tension control and porosity of the sheet undoubtedly govern the coating weight to some extent although it is claimed that from 6 to 60 lb. per ream can be deposited.

There are a host of other patents that cover a coater of one type or another which has been specifically designed for polyethylene and poly-wax blends or can be easily modified to handle these materials. However, almost universally these methods do not apply a premetered amount of coating and would, therefore, be no more accurate than the caliper variation of the paper or board being coated. Since we feel it is vitally important to maintain accurate coating thickness, primarily because of high cost of resins, a method was decided on that had this feature.

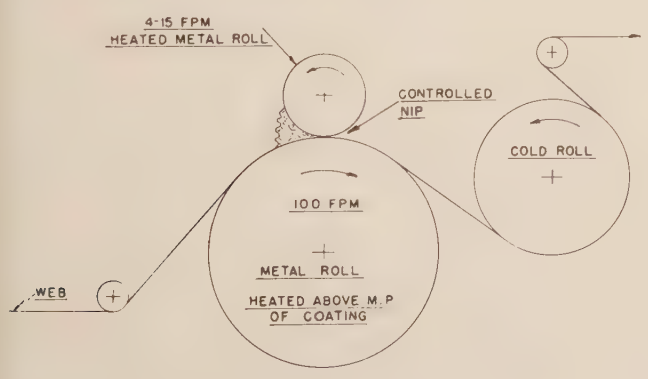



*Fig. 5. A. Abrams, pat. 2,089,524*

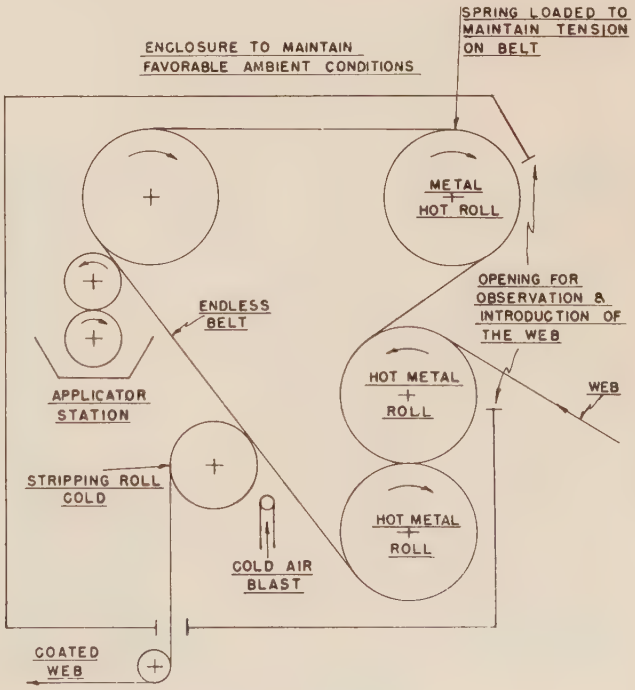



*Fig. 6. J. W. Little, pat. 2,559,649*

This method was reverse roll coating. In addition to applying only a premetered amount of coating, a reverse roll coater is excellent for handling relatively high viscosities. The main bulk of development work in roll coating the medium viscosity range of polyethylene and polyethylene-wax blends during the past two years has been done with a four-roll reverse roll coater called "Contracoater"* (*11, 12, 24, 25*).

### *Reverse Roll Method*

The Contracoater is shown in Fig. 8 with an electrically heated oil-jacketed pan. Electrical tubular heaters were used in the three rolls as shown.

Note the direction of rotation of the rolls in Fig. 8. It will be seen that the rubber backup roll, applicator roll, and metering roll all revolve in the same direction. The pickup roll revolves opposite to the others in order to form a premetering nip with the applicator roll. In operation the speed of the pickup roll is $^1/_5$ to $^1/_{20}$ of the applicator roll. The metering roll runs from $^1/_5$ to $^1/_{50}$ the speed of the applicator roll. Last, the applicator roll may run from $^1/_2$ to 5 times the speed of the rubber roll (web speed). The relative speeds of the rubber backup roll and the applicator roll are known as the wipe ratio. For instance, if the applicator roll is running twice as fast as the backup roll, we have a wipe ratio of 2:1.

For the polyethylene work, a fifth roll was added for cooling means as shown in Fig. 9 (*13*). This work was carried out for the purpose of developing a machine and process to handle the "lost viscosity range," i.e., a range too heavy for conventional roll coating machinery, and too fluid to form a film in the extrusion lamination method. It has been found that the reverse roll coater has a top range of 25,000 to 30,000 cp. viscosity in handling polyethylene and polyethylene-wax blends. This blend range and polyethylene might be considered the lower practical range for extrusion too. In other words, the reverse roll coater and extruder tend to complement each other in handling these blends. The upper practical range of temperature appears to be about 350°F. for these materials in roll coating. Above this temperature, oxidation is apt to increase rapidly since oxidation increases with any increase in temperature. It is interesting to note that the upper limit for this type of coating applied by roll

* Trademark, patent pending.

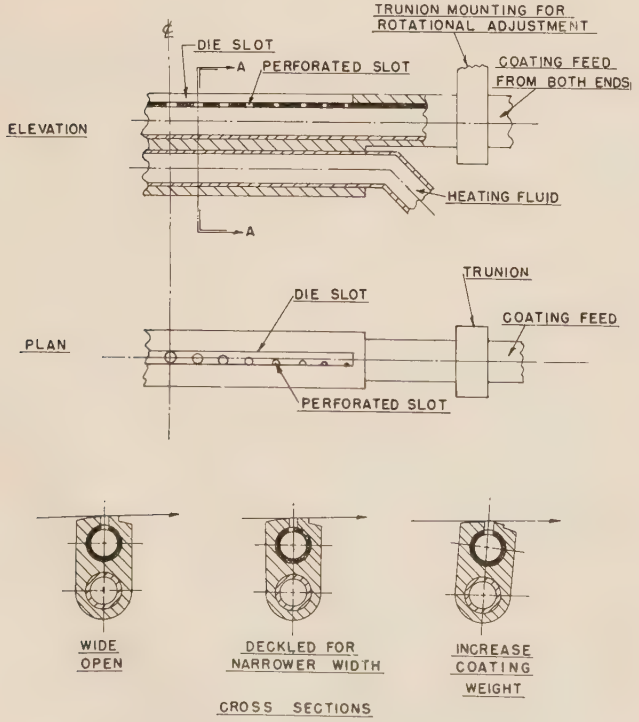



*Fig. 7. L. J. Van Guelpen, pat. 2,464,771*

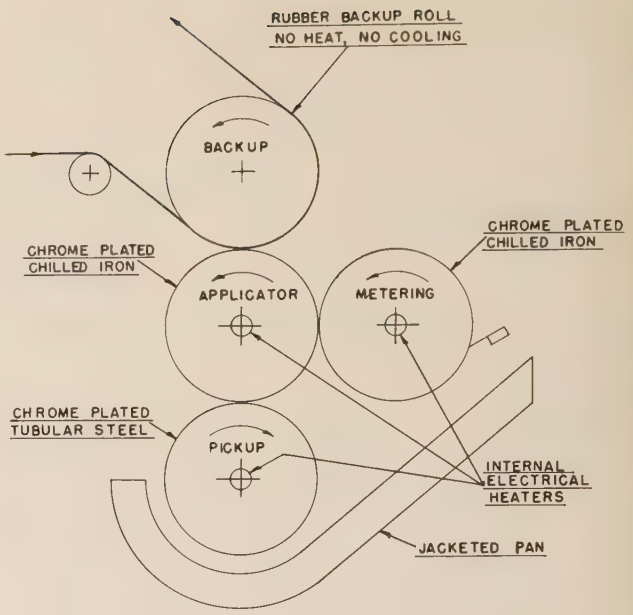



*Fig. 8. Contracoater four-roll reverse roll coater*

coating was recently described by Burgess and Swec to be about 300°F. and 10,000 cp. viscosity (*14*). No indication was given as to the type of equipment on which this evaluation was made.

Pinhole-free films of polyethylene have been coated with as little as 10 lb. per ream (3000 sq. ft.). With the poly-wax blends, it has been found necessary to deposit approximately the same amount to achieve pinhole freedom. No conclusive difference has ever been found in moisture vapor transmission rate of poly-wax blends that were roll coated as compared to extrusion coating of the same blends. One big advantage of roll coating that has been found is the adhesion of the film to the web. In no case has it been possible to remove the cast film without a paper tear when roll coated (*13*). This is undoubtedly because of the method of application, that is, reverse roll coating.

We have taken samples of photomicrographs of the roll-coated film using a blend of 60% polyethylene–40% wax and samples of the same blend extruded in order to study the surface characteristics and lamination of a blend to the paper.

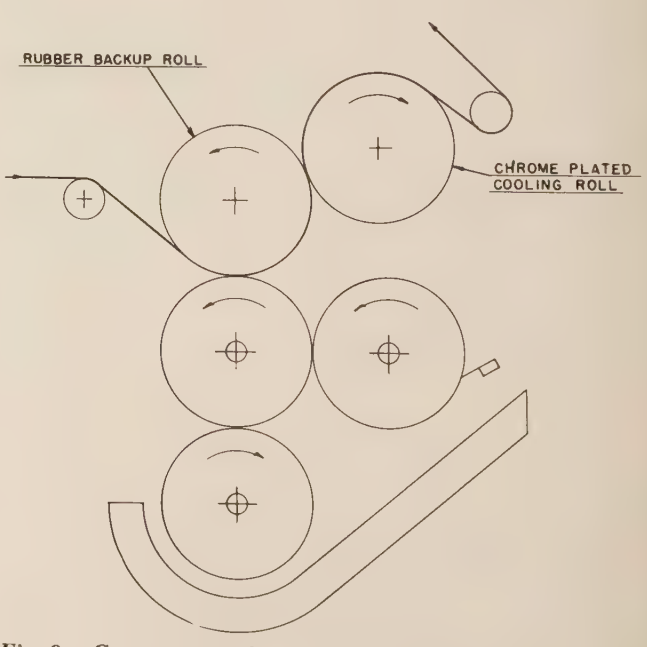



*Fig. 9. Contracoater four-roll reverse roll coater modified with a cold roll*

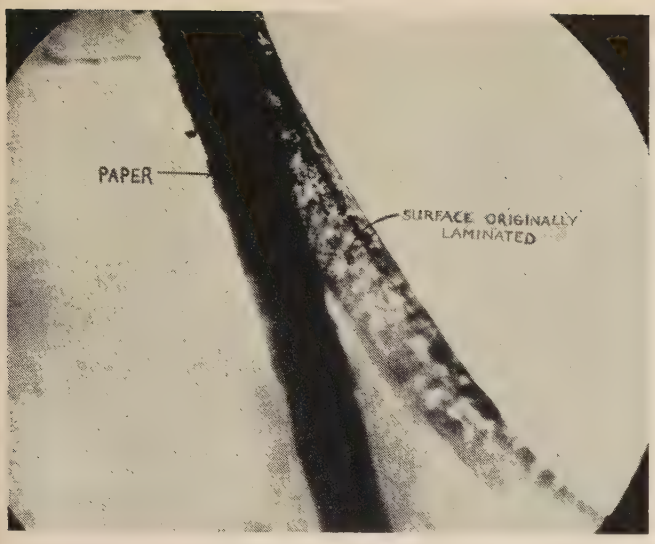



*Fig. 10. Extruded*

Although definite proof has not been found by photomicrograph study, it would appear that adhesion of coating to the web by roll coating is superior. Figure 10 shows a photo of the extruded blend on paper. Note the way in which the film has separated in the extruded sample and also the apparent lack of intimacy between the film and the fiber.

In the roll-coated sample shown in Fig. 11, the film is still firmly attached and appears to be very well anchored.

The highest speeds achieved to date have been 200 f.p.m. on 60% poly–40% wax having a viscosity of 28,000 cp. at 350°F. This has been done in a pilot plant (*13*). It is thought that the top speed will be somewhere around 600 f.p.m. under normal conditions. If the "necking down" or stretching properties of some blends can be employed, this speed can be increased by virtue of a slower speed on the applicator roll than the actual web speed. Of course, the surface condition of the web will govern this to some extent. For instance, to obtain pinhole freedom on board, it is usually necessary to employ a wipe ratio of 2:1 or more because of the rough surface.

The chill roll that was shown in Fig. 9 is vital. This roll must be under pressure against the backup roll to achieve a smoothing of the coated surface, i.e., remove the stipple pattern, and to develop the optimum gloss. It has been found that some of the irregularities of the coated film can be rectified by this roll through control of the temperature and pressure. Machine speed has a large part to do with the conditions under which this cold roll operates best. In some trials, it was found that the gloss development was equal to that achieved in extrusion but because of incomplete chilling throughout the coated thickness, a mottled coating developed after standing for a few hours (*15*).

Photomicrographs were taken to study the surface characteristics of extruded film versus roll-coated film. No real difference has been found consistently as yet although further study and improvement of sampling and staining techniques may soon give us more information.

It has been found that some webs should be preheated to drive off excess moisture. If this is not done, the vapor thus formed will blow through the coating and cause pinholes. Further, this vapor disrupts the intimacy between the molten coating and the chill roll thus giving the appearance of "shiners" in the coating since the intimacy necessary is obtained only where these holes have raised the coating. This phenomenon has occurred only on board to date (*5*).

The heating of coating pan and rolls is extremely important in the reverse roll coater. The best condition we have found is to have the pan about 75 to 125°F. above the melting point of the resin and the rolls about 15 to 25°F. cooler than the coating in the pan. The reason for the latter is as follows. Upon referring to Fig. 12, it will be seen that the opening between the applicator and metering roll is 0.005 in. or $^1/_2$ mil. The cast iron roll will contract or expand 0.00004 in. with a 1.8°F. temperature change. If the rolls are held at the same temperature as the coating, they will be in danger of actually crashing together under operating conditions since the center of the applicator roll tends to cool down from being contacted by the web. Upon losing heat in the center, it is then necessary to move the metering roll closer to the applicator roll to maintain the 0.0005-in. opening. Assuming the rolls and bearings were perfect, there would have to be a 22.5°F. change between the ends of the rolls and the center to have the ends in contact and an 0.0005-in. opening in the center. Unfortunately, rolls and bearings are not perfect. With a coater designed as the Contracoater is, having horizontally mounted accurate rolls, the practical accuracy built into the machine is about 0.0002 in. additive runout which will reduce the maximum allowable temperature variation to 13.5°F., before contact could occur. Therefore, we have found to preclude this difficulty that the rolls should run colder than the coating by an amount sufficient for the excess heat in the coating to replace the heat dissipated from the applicator roll to the web and rubber roll.

It is interesting to note that a three-roll reverse roll coater using a similar method of chilling a hot cast film was recently described by Sturken and Egan (*16*). It was found that with an extruder feed, as shown in Fig. 13, pure polyethylene with a molecular weight of 14,000 could be applied in thin and continuous films. No indication was given as to the temperature of the polyethylene although the literature would show this particular polyethylene to have a viscosity of about 30,000 cp. at 325°F. (*17, 18*).

## EXTRUSION LAMINATION

The photograph shown in Fig. 14 is the installation of a polyethylene extrusion laminator from the winder and taken from above the machine. At the far end is the continuous Kohler† Unwind Reel featuring running belt tension. Next in line is the extrusion-lamination section. Not shown in the photograph is a Mount Hope spreader roll to level the sheet prior to entry into the laminating section. Following the laminating section is a slitter section to trim off the bead and uncoated paper. Beta ray gages were added to control caliper of the coating on the sheet.

From the slitting section, the web goes to the continuous Kohler† winder.

† Trademark, patented.

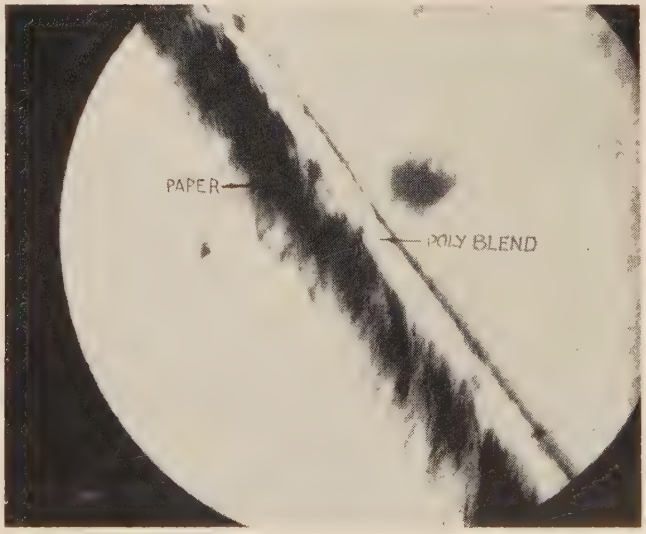



*Fig. 11. Roll Coated*

Table I. Typical Extruder Data

| Extruder size, in. diam. | Heat on barrel, watts | Effective screw length, in. | Floor dimensions, in. | Weight, lb. | Capacity, lb./hr. | Drive, hp. |
|---|---|---|---|---|---|---|
| $1^1/_4$ | 4,800 | $21^1/_2$ | 47 × 26 | 1200 | 15–20 | 2 |
| 2 | 10,500 | $31^1/_{16}$ | 85 × 33 | 2900 | 60–70 | $7^1/_2$ |
| $3^1/_4$ | 19,400 | $44^3/_4$ | 120 × 37 | 4000 | 130–150 | 20 |
| $4^1/_2$ | 35,000 | $60^5/_8$ | 130 × 41 | 5550 | 250–300 | 40 |
| 6 | 48,000 | $79^{11}/_{16}$ | 131 × 81 | 7200 | 500–600 | 50 |
| 8 | 75,000 | $91^5/_8$ | 144 × 108 | 8000 | 800–1000 | 75 |

### *Machinery and Methods*

There are many bulletins, articles, and books published today explaining in detail the general operation of extrusion laminators, so we will not repeat that which has been published (*17, 18, 19*). However, there are generally three major fields of importance in a polyethylene extrusion laminator machine. First, it is very important to have continuous operation, or web flow. This is to prevent having to shut down the machine for roll changes.

Second, is the extruder and die. Satisfactory operation of this component is obvious. Usually the feed screw is of uniform pitch and decreasing depth, with a metering section at the end to help stabilize the pressure upon the die (*19*). Once this component is operating satisfactorily, it should never be shut down except in case of emergency, or when the run is completed. The reason for this is that it may become necessary to clean the die of oxidized polyethylene unless the heat on the extruder is shut off or very carefully controlled. Then, too, time and materials are lost getting the machine in adjustment after the start-up.

There are means provided in the die to adjust the die opening to compensate for any caliper variation occurring in laminated film. The die is adjusted with a sectionalized backing plate that loads against the die lips. Usually, the opening between the lips is about 0.015-in. with the desired film thickness achieved through variation of the web speed with respect to the extruder screw speed and the temperature of the molten resin. The extruders are usually built in a range of sizes from $1^1/_4$ to 8-in. diameter screw. The corresponding capacities are 15 to 1000 lb. per hr. Table I gives the specifications for a typical range of extruders (*20*).

The third component is the lamination section which consists of an air-loaded rubber-covered pressure roll around which the web is wrapped and a cooling drum on which the cast polyethylene is laminated and chilled. The heart of the unit is the cold roll. It should have a highly polished surface in the case of most uses since it is transferred to the polyethylene film as a mirror image. However, there are many times it is desirable to have a satin or velvet finish on the sheet for better slip and bag forming characteristics so it is then necessary to use a chemically etched roll or a roll having a roughened surface. It must have sufficient cooling capacity for the maximum production from the extruder and at the top speed of the machine. Many different designs of cold rolls have been proposed and are in use today. One of the most efficient is that shown in Fig. 15.

The engineering fundamentals behind the design are easily explained in that a high velocity stream of coolant is directed in a confined manner and in helical fashion around the inner periphery of the drum to achieve a turbulent flow of coolant and to scrub the inner surface of the roll. The biggest deterrent to heat transfer is the film of scum and scale, called the fouling factor, that builds up on the surface through which heat must flow. Thus, if the layer is kept at a minimum, the heat transfer will be good. Further, in this design, the inner shell is removable so that access can be gained to the inside of the forging to clean the inner surface periodically. This particular patented design is known as the Gross Heat Transfer Insert and has been used successfully for years in rubber mills to cool rolled sheets (*21*). Tests have shown this cooling drum to be about 30% more efficient than other double shell roll designs under most operating conditions (*22*) Fig. 16.

### *Poly-Wax*

Of great interest today in extrusion is the extrusion of poly-wax blends since straight poly extrusion is now quite well known. Experimental runs have been reported with blends having as much as 40% paraffin (*23*). It is necessary to maintain sufficient pressure on the die through use of lower temperatures on the extruder and a finer screen pack when extruding blends. The greater the amount of wax, the more critical is the temperature control since viscosity of the blend is directly related to the pressure on the die, and with ever increasing amounts of paraffin, the more the extruding range approaches the steep portion of the curve of viscosity versus temperature of these blends. This was shown in Fig. 2.

Adhesion is of great concern when using poly-wax blends. Since it is sometimes necessary to work only 80 to 120°F. above the softening temperature of these blends, there is not sufficient heat in the blend to give the adhesion that is normally achieved with straight polyethylene. The adhesion problem becomes more pronounced with a greater portion of wax because the greater the amount of wax, the lower is the

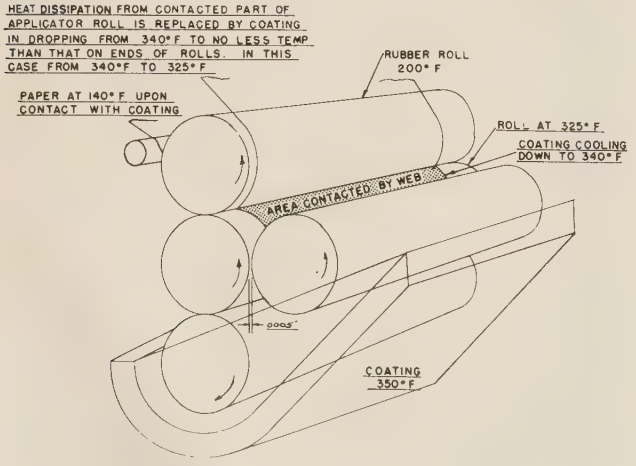

HEAT DISSIPATION FROM CONTACTED PART OF APPLICATOR ROLL IS REPLACED BY COATING IN DROPPING FROM 340°F TO NO LESS TEMP THAN THAT ON ENDS OF ROLLS. IN THIS CASE FROM 340°F TO 325°F
PAPER AT 140°F UPON CONTACT WITH COATING
RUBBER ROLL 200°F
ROLL AT 325°F
COATING COOLING DOWN TO 340°F
AREA CONTACTED BY WEB
.0005"
COATING 350°F

*Fig. 12. Roll distortion from heat*

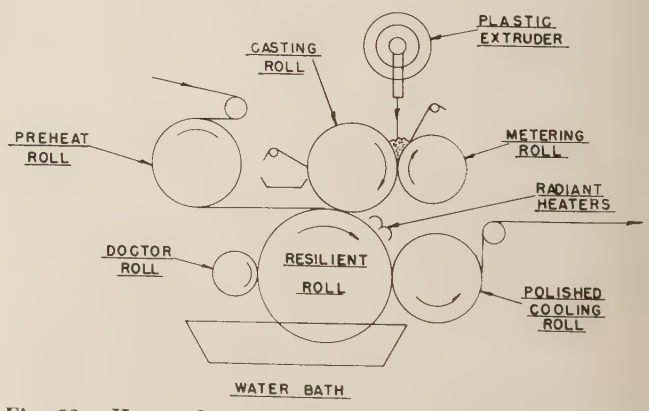



*Fig. 13. Hot melt reverse roll coater with extruder feed*

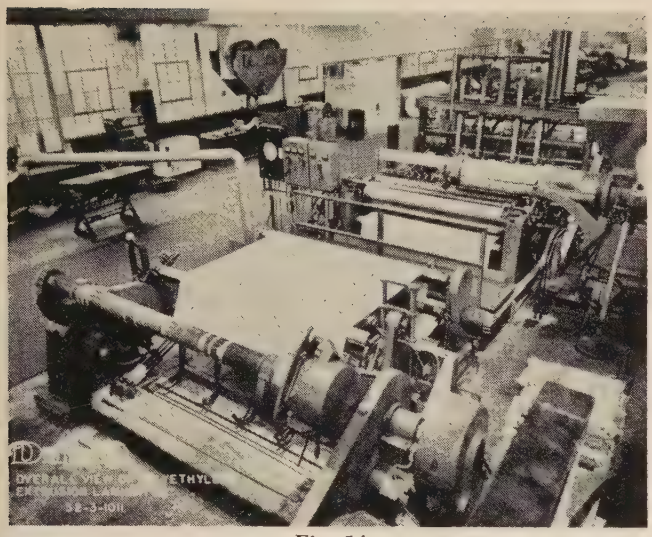


*Fig. 14*

temperature of extrusion. To obtain the adhesion, it has been suggested by some sources to preheat and then postheat the substrate. Another critical control for the adhesion problem is the chill roll temperature. It is necessary to approach the temperature at which the extruded film will adhere to the chill roll. Normally, this is about 165°F. (*23*).

## CONCLUSIONS

To sum up the relative merits of the roll coating and extrusion-lamination methods of applying polyethylene and polyethylene-wax blends, the merits of each will be discussed first on polyethylene and second on the blends.

### *Polyethylenes*

It should be obvious that each method is limited in its scope due to viscosities of the various polyethylenes. It can be stated with some degree of accuracy that roll coating methods are good for polyethylenes 12,000 molecular weight and under, and/or viscosities up to about 25,000 cp., at temperatures not over 350°F. Extrusion is normally used for polyethylenes having a molecular weight of 19,000 and over, and can handle melts up to about 600°F. When working in the range of viscosity at which either method was applicable, the choice of equipment would be governed by the products to be manufactured and the particular polyethylenes to be used.

### *Polyethylene-Wax Blends*

For poly-wax blends, the normal breakpoint, or common

*Fig. 15*

range would be about 60% poly (12,000 m.w. and up), 40% wax. If adhesion were critical, it might be desirable to roll coat. Of course, the same reasoning as just mentioned should be applied in any case, in that the final choice of equipment would be governed by the products to be manufactured and the resin formulations that would most regularly be used.

A very strong point for the use of a roll coating method in preference to the extrusion-lamination technique is that no edge bead is left. A coating can be carried out to the edge of the sheet with no loss of coating or paper. It has been reported that there is considerably more necking down with the blends, and would, therefore, tend to magnify this problem.

Converters and machinery manufacturers have been attempting to eliminate the bead problem by wetting the rubber pressure roll with water extruding beyond the web and trimming of poly, but to date, this has not been universally successful. If the type of base stock changes frequently, i.e., foil, scrim, paper, cellophane, and others, then a different amount of water has to be applied behind the web on the rubber pressure roll for each type of web In some cases, the rubber roll should be warm, in others cold. Then, too, it is necessary to have a different amount of water on the edges where the film contacts the rubber roll than the amount of

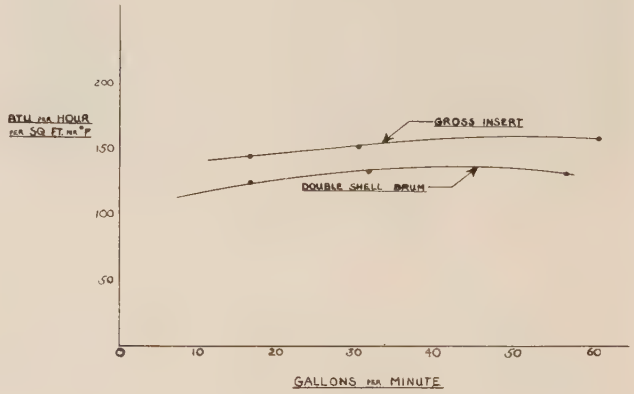



***Fig. 16. Tests made on a 30-diam. by 48-in. wide forged steel roll running at 400 f.p.m.***

water in the center. To perform the metering operation by squeezing two rubber rolls together is all right, but the water addition cannot be made greater on the edges without special grinding of the rubber roll. Then, when the web width is changed, the whole metering arrangement is thrown off. The method of extruding beyond the sheet can be made to work, but not without elaborate equipment and compromises. The bead problem is serious when consideration is given to the unsalvageable waste of base stock and polyethylene. Trim is sometimes 2 in. wide.

## CONSIDERATION OF METHODS

In closing, there are two final thoughts to consider in the choice of products to manufacture and equipment to purchase. First, is the question of continuous operation. To use continuous operation in extrusion lamination machinery is desirable because of the economics involved. The continuous unwinding and winding stands will add to the relatively high initial investment. Then, it will be necessary to run straight polyethylene or polyethylene-wax blends that are predominantly polyethylene. The cost of resin or blend will range from $0.30 to $0.43 per lb.

If roll coating is used, it is not necessary to use continuous operation since the coating machine can be stopped for short periods of time with no serious effect. The initial investment in equipment is lower than that necessary for extrusion lamination, and has the further advantage of being able to handle polyethylene-wax blends costing from $0.25 to $0.35 per lb. Therefore, the second consideration is the cost of the final prod-

*G. E. Rabeler and Paulene Chu, International Paper Co., Junior Award Winners*

uct based on machine amortization, raw materials cost, conversion cost, and overhead. Obviously, roll coating does have many advantages for the new field of coatings that are developing in polyethylene, and should tend to complement the extrusion method used today.

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## Modification of Plastic Films for Specific Packaging Needs


*James B. Sisson*


THE title of this paper is limited by the words plastic film and packaging. Plastic film refers to a continuous material which is flexible, self-supporting and less than 3 thousandths of an inch thick manufactured from a group of synthetic chemicals now commonly referred to as plastics. Packaging implies the use of the film functionally to contain and protect materials and otherwise to display, enhance the appearance of and to identify the contents.

Identification and enhancement of a package involve printing and coloring. These are modifications of a plastic film and hence come within the title. However, I felt that the printing field is so large as to require separate treatment so in the remainder of the paper only a few references to printing will be made. Those of you concerned directly with this business cannot so conveniently forget the printing phase, however.

We will consider five different methods of modifying films which apply to their containing and protecting functions. These are:

1. The method of laminating a film to a base sheet of different material such as paper, cloth, foil, or cellophane.
2. The method of coating one film with another functional coating.
3. The method of copolymerization.
4. The method of blending the film base with extenders and plasticizers.
5. The method of creping, embossing, stretching, or otherwise physically altering the film to change its properties.

One specimen may often involve more than one of these types of modification.

The first method is of the most interest to the paper industry because it includes plastic coated papers. Why laminate a film to paper? Paper is the material which offers more strength per dollar in a flexible sheet than any other. Since strength is an important packaging requirement the strongest type of paper, kraft, is an important web material to which films are laminated.

A 50-lb. natural kraft with 15-lb. polyethylene is used in multiwall bags for the purpose of containing and protecting materials which take on water from the air rapidly, such as calcium chloride, or which may rapidly attack paper, such as acid phosphate concentrates.

A 42-lb. bleached, wet-strength kraft with 8-lb. of polyethylene coating is used as a frozen meat wrapper. The flexibility at low temperatures and water vapor barrier qualities of the film are combined with the strength of the paper to form an excellent single-ply wrap for frozen meat products.

The requirements of a pouch paper for a number of food, drug, and soap products such as sugar, moth crystals, and soap powder include moisture resistance, flexibility, heat sealability, and reasonable strength. Bleached paper provides a satisfactory printing surface. The construction is 30 lb. of bleached kraft with 6 lb. of polyethylene.

In the foregoing instances the film thicknesses have been one thousandth of an inch in the first case and about $^1/_2$ thousandth in the last two. At least $1^1/_2$ and usually 2 thousandths of an inch of polyethylene film unsupported by paper are required for packages.

Another form of paper is used for quite a different reason.


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I refer to glassine which is a nonporous cellulose sheet produced mechanically. True glassine is an excellent gas and grease barrier but it usually is relatively weak and brittle. Polyethylene is flexible and a good moisture barrier but although not chemically affected by gases, organic solvents, and oils is not classed with cellulose as a barrier to them. Glassine laminated to polyethylene constitutes a barrier to a very wide range of materials which has improved strength qualities over either component by itself.

Cellophane laminated to polyethylene provides a case very similar to glassine except that the greater transparency makes the use of this combination more desirable in many cases. This combination is one of the newest on the market and its range of barrier and strength qualities is one of the widest in a transparent flexible material.

Cellophane normally is moistureproofed with a lacquer composed of nitrocellulose, wax, and natural resin which has very good moisture resistance in the flat state but which suffers substantially when creased. The flexibility of polyethylene overcomes this difficulty.

If seeming overemphasis has been placed on polyethylene it is for two reasons: (1) I work with it; (2) it is the fastest growing plastic and has the rosiest future of any thermoplastic now commercially produced.

There are other plastic films surely which are important and one of the most versatile of these is polyvinyl chloride or more commonly its copolymer vinyl chloride-vinyl acetate. It is tough when plasticized, hard when not plasticized. It is moisture resistant and resistant to all but a rather small group of solvents. With it you can make rigid pipe, flexible pipe, all manner of moldings, and film, either solvent deposited or extruded. It has good heat seal properties. Currently, the yard swimming pool is probably the most colorful use of this class of compounds which are usually referred to as vinyls.

Vinyls, too, gain value in some cases where they are laminated to other webs. One of the most interesting and progressive developments in the paper field has been the vinyl-coated board for milk bottles—nestile containers made by solvent deposition of a thin vinyl film on each side of a stiff semibleached kraft food container board.

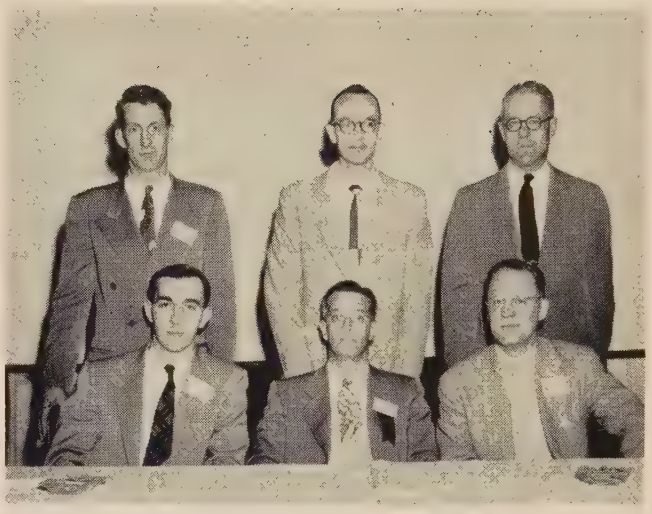
Converting machinery developed at the same time makes it possible for a dairy to receive sidewall blanks, tops, and bottoms all shipped flat to save space. The machine converts these automatically into a bottle, fills it with milk, and seals a top on. If these few words make it sound simple, be assured that it isn't.

Another case of improvement by lamination is that ultimate in packaging, the foil laminate. In creped kraft, aluminum foil, and vinyl film the paper is for strength, the 1-ml. aluminum foil is a perfect barrier when flat and pinhole free, and the vinyl film provides the foil with a tough scuff-resistant surface capable of forming perfect heat seals which are adequately moisture vaporproof. This material is designed to meet certain classes in JAN-P-131 government Specifications for Barrier Materials, Flexible, Moisture Vaporproof.

Other plastic films such as cellulose acetate, vinyl acetate, and numerous rubber latices are laminated to paper for similar reasons. To consider all cases would occupy too much time. My chief objective is to acquaint you with the principal methods of film modification.

The next method and certainly a relatively old one is to coat the film with another material.

A coating process to make cellophane moisture resistant was developed in 1927. Today a major portion of cellophane used for packaging is coated. The coating itself is lacquer composed of wax, nitrocellulose, natural resins, and solvents. It is deposited in a film of about $^{1}/_{10,000}$ in. in thickness on the surface of the cellophane. The moisture vapor barrier qualities of flat cellophane so treated are similar to comparable weights of polyethylene. However, crease resistance is not as good. This coating can be heat sealed but the heat seal formed is weak in a moist atmosphere.



*Seated: G. L. Booth, The Black Clawson Co.; J. D. Parmele, St. Regis Paper Co.; and B. W. Weiner, E. I. duPont de Nemours. Standing: H. T. Holland, Monsanto Chemical Co.; J. B. Sisson, St. Regis Paper Co.; and S. Orton, Jr., Orton Corp.*

Efforts to solve this problem finally resulted in a patented anchor coat of a wet-strength resin such as urea formaldehyde. When the moisture resistant lacquer was applied over this anchor coat, heat seals made subsequently were stronger and held over a much wider range of atmospheric conditions.

Letters are used to designate cellophane qualities. One containing all the foregoing modifications would be denoted by the letters MSAT—moisture resistant, sealable, anchored, and transparent.

Experimental work has been done with polyethylene coated paper which indicates that substantial improvement in grease and gas resistance of polyethylene can be accomplished by overcoating with grease and gas resistant material.

Pouches of overcoated polyethylene have been developed experimentally which will retain turpentine for 100 times as long as uncoated polyethylene. Such a material, to my knowledge, is not at present commercially available however.

A third method of modification is copolymerization. High polymers are formed from materials which have two or more reactive groups attached to each molecule. When such a case exists and enough energy is supplied in terms of heat or pressure in the right medium and with proper catalysts, these units can join together much as a group of people can join hands to form chains. In many cases it is possible to modify the polymer by introducing amounts of a second and even a third monomer which also have two or more reactive groups per molecule.

One of the most interesting cases in the packaging film field is Saran which is a copolymer of vinyl chloride and vinylidene chloride. This material as a barrier has just about everything that a combination of cellophane and polyethylene has except ease of manufacture. Saran does not lend itself to solvent coating and it rapidly decomposes at extrusion temperatures. Film manufacture is therefore a rather difficult procedure. Printing is also difficult. Despite these obstacles considerable quantities of this material enter the packaging fields, such as a household refrigerator food wrap or bowl covers.

A second widely used commercial copolymer also has vinyl chloride content. It was the one originally mentioned in connection with the vinyl coated board and the vinyl coated foil paper laminate. It is the vinyl chloride-vinyl acetate copolymer.

Other copolymers are of considerable commercial importance as plastics such as cellulose acetate, butyrate, styrene

butadiene, and styrene-acrylonitrile. Thiokol-epoxy and polyamide-epoxy resins are new thermosetting resins which can form flexible coatings. The future of copolymers is highly promising. However, copolymerization is limited quite definitely to the resin manufacturer. Another somewhat similar method available to the converter is the use of extenders, plasticizers and other additives to change the properties of a plastic film.

The use of plasticizers in film materials has been common since the 1920's when cellophane was, and to a large extent still is, plasticized with glycerine. The objective successfully accomplished was to make the cellophane pliable and tough rather than brittle.

Vinyl chloride and vinyl chloride acetate copolymers are usually plasticized as films. A large number of plasticizers for these materials fall into the category of high molecular weight esters. Tricresyl phosphate, butyl sebacate, methyl phthalate, tributyl citrate are examples of effective plasticizers. The choice of one over the other depends upon the expected atmospheric conditions of use, whether or not toxic materials are tolerable in the end product and without doubt —cost.

Cellulose acetate film is another which requires a content of good plasticizers such as triphenyl phosphate to perform effectively.

The plasticizers referred to thus far are liquids at room temperature. Although they are effective and will have continued use they are not ideally permanent because of their tendency to bleed and to evaporate leaving a brittle, easily broken film.

Some solutions to this problem are available for certain plastics. More flexible high impact polystyrenes are prepared by blending a synthetic rubber with the rather brittle polystyrene. A material plasticized with nonvolatile elastomer is in general more permanently flexible and tough than one plasticized with a liquid even if its volatility is very low. Considerable work remains to be done along this line with the cellulosics and vinyls.

Extenders are also used with plastics. Natural cumarone-indene resins are compatible with polystyrene. Waxes are compatible with vinyl chloride and polyethylene in varying degrees. Butyl rubber and Vistenex or polyisobutylene are compatible with polyethylene as are some petroleum resins.

An extender is used in a plastic to maintain desired properties at lower cost. One of the most promising applications of an extender is the case of wax with polyethylene. Forty parts of 10¢ a lb. microcrystalline wax can be blended with 60 parts of polyethylene at 40¢ per lb. to yield a flexible moisture resistant extrusion coating material at a reduced cost. It has been recently demonstrated that amounts of petroleum resin or synthetic rubber can be added to such a blend to make possible even lower polyethylene contents in an extrusion coating and still lower costs. Such a coating is flexible but harder and more temperature resistant than most wax-rubber blends. It is however, less resistant to solvents than polyethylene.

The last method of plastic film modification which I have considered is that of physical change. The effective packaging efficiency of a coated paper may be improved for some applications by off machine creping to provide stretch in two directions.

Another possible physical treatment is surface etching or embossing usually done in the coating process with press or calender rolls. Less tack to sticky materials, better slip against forming surfaces, and a nonglossy surface are possible results which may be desirable.

Films may be stretched to orient them in one or two directions. Such films are usually stronger and/or more flexible than solvent deposited unoriented films. One grade of Saran film is stretched in two directions and at the same time cooled to set it. This product is used very uniquely and effectively for pouches for turkey and chickens. The turkey is placed in the pouch and then dipped in boiling water. The two way stretched Saran film tries to regain its original dimensions and shrinks tight around the fowl to form a second moisture resistant skin.

A recently patented flame treatment for polyethylene film provides a "tooth," either chemical, physical, or both which permits some types of printing inks to key to it effectively.

One of the newest and still developmental semiphysical processes for altering polyethylene is to expose it to high-voltage x-rays. The irradiated polyethylene is chemically altered to become a material with a much higher melting point and distortion temperature. Such means of continuing polymerization may find a place in the synthesis of other plastics.

This completes this rather short presentation of the subject. I have attempted to provide you with examples of five general methods of altering plastic films. These are again briefly: Lamination—films are applied to cloth and paper webs for strength and to glassine, other films and foil for improvement in barrier qualities. Coatings—coatings are effective means of adding barrier and heat sealing properties to films. Extremely thin film coatings in the order of one $^1/_{10}$ of a mil thick can bring about 100-fold changes in barrier properties in some cases.

Copolymerization—This chemical modification of plastics is very effective in giving a material just the right blend of properties for a job.

Allied with this are the effects gained by mechanically blending additives with plastics. Most plastic films require plasticizers to render them flexible. Occasionally an additive may improve a barrier quality. Many additives are extenders used to maintain a barrier effect at lower cost.

Physical modifications of film are varied in nature and effect. Film orientation can markedly improve the strength and flexibility of a film. Surface properties of a film are substantially varied as the film goes from an etched to a glossy surface.

Special effects may be obtained by heat treatment or irradiation. If this short review of film modification methods brings to your minds a possibility for attacking one of your problems it serves its purpose.

## Foam Measurement

*Hugh T. Holland*

THE interest of Monsanto Chemical Co. in paper mil foam measurement is that of a supplier of raw materials which do not cause foam, rather than as a supplier of chemical defoamers. As you might expect, coordinating laboratory and mill results on foam measurement is a difficult problem. In the past, judging by actual mill trial was very deceptive because a successful run in one mill proved little when a new run was planned. The industry catchall attributing discrepancies to local mill conditions was offered. This excuse was not too well thought of since the deeper an investigation gets on local mill conditions, the less truly basic differences are found. To shorten the history, let me say that a concentrated effort by our laboratory and some excellent cooperation by mills of the paper industry gave us the groundwork to devise numerous methods possessing some merit. However, it was not until a correlation of foam action and types was made with mill operations that the present test with proved practical reliability was adopted.

Paper mill foam is of two basic types, easily recognized: (1) large bubbles which can be broken by showers, sprays, or surface-active defoamers and (2) very small bubbles adsorbed or attached to the individual fibers. This attached foam is by far the more troublesome and sometimes defies the most efficient chemical defoamers. Occasionally large

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volumes of visible surface bubbles in the system cause consternation throughout the mill. However, corrective action sometimes only results in making little ones out of big ones. Unless the amount of foam is considerably reduced the trouble has just begun. These small attached bubbles cause the beater to overflow and reduce beater circulation. Next they cause stock pump binding from the beater chest or produce uneven consistency due to heavy and light stock. Similar action is seen at the machine chest while on the machine these small bubbles are responsible for fiber flocculation in the headbox, a decreased drainage rate on the wire, and lastly, they trademark the sheet with the characteristic, circular thin spots or free spots formed in the sheet as they are broken at the flat boxes. We have found that materials which stabilize this attached foam are the most troublesome in paper mill operation.

Consequently, a valid test must measure the relative amount of attached foam produced under standard test procedures. The methods of measurement vary. The preparation for a test we have found to be valuable consists of aerating a stock-water-size mixture with an egg-beater-type mixmaster, then adding alum with further aeration. The resultant slurry is then poured into graduates up to the 250-ml. mark and is allowed to stand. The foam bubbles attached to the fibers and trapped in the pulp cause the fiber and water to separate, the degree of flotation being relative to the foaming properties of the mixture. Another method of determining the relative foaming properties is to measure the amount of entrapped air. This can be done by evacuating or, more simply, by adding defoamer to release the air, then noting the decrease in volume.

Although the primary interest in this test is for measuring the foaming properties of internal sizes, we consider that this test procedure is applicable to other constituents of the furnish. One interesting experiment consisted of determining if any specific grade of wastepaper was especially troublesome in the mill. All the grades used were tested until the trouble-maker was found, then by further testing the maximum quantities of this grade usable without severe foam troubles were determined. The results agreed satisfactorily with later mill experience. In another instance, work on foaming constituents from sulphite waste liquor showed a straight line function when plotting per cent air volume versus concentration.

The test can also be used to measure the relative effectiveness of chemical defoamers at various levels of application.

In conjunction with this standard laboratory foam test, we have attempted mill measurement of air in the stock at the headbox on fourdrinier machines in order to develop quantitative figures on machine foaming conditions. Numerous tests have been made by TAPPI Routine Control Method No. 173, in which the foam measurement is based on the weight difference of a special tube filled with headbox stock before and after deaeration. The sample used runs about 250 grams in 400 grams of glassware and contains air displacing about 1 gram of water. This makes measurement of small changes secondary to technique. Consequently, I would prefer to modify the test somewhat before recommending it for general use.

The most successful test of operating conditions to date has been the measurement of the tendency of headbox stock to float, just as in the laboratory test discussed previously. The major advantage of this test is that it gives a larger range so that changes in operating conditions and furnish are more obvious. I recall one instance where the numerical value of this test nearly doubled when a machine tender diluted his defoamer solution rather than make up a new batch at a shift change.

It appears that a combination measurement of the degree of flotation and volumetric determination of the amount of air will provide the required accuracy of test to give a practical means of determining quantitative foam in paper machine operation.

We are all aware of mechanical means of eliminating foam, such as the Deculator and Vortrap Degasser and of the chemical means by the use of defoamers. When necessary, these do a thorough and satisfactory job. However, it is felt that guided by a test of the type we have been discussing, much can be done within the mill to improve or correct poor operating conditions, such as pumps sucking air and unnecessary aeration of stock through cascades and high turbulence flows, as well as singling out troublesome components in the beater furnish. In most cases, correction of these troubles will result in a real improvement in quality and cost.

In summary, I would like to go over the simple principles and procedures involved: (1) the effect of the attached air bubbles is most important and is the proper basis for a test, and (2) measurement can be either through the buoyant effect on the fiber or by a determination of actual air volume. I want to emphasize that the laboratory foam test has proved to be a reliable indication of results to be expected in the mill but we have found no completely satisfactory mill test for control use at the machine.

## Applications of the Hurletron Automatic Caliper and Basis Weight Control to Paper and Paperboard Machines

*Carl M. Noble*

Although applied principally, to date, to the manufacture of boxboard, the present caliper control equipment (Hurletron Model MG-649) and modifications thereof are applicable to gaging and controlling the thickness of fine papers, newsprint, laminates, plastic sheets and films, coated stocks, insulating board, wallboard, hardboard, asbestos-cement, gypsum and chip boards, and similar nonmagnetic materials produced in sheet form. Adaptations of the system can also, conceivably, be applied to either magnetic or nonmagnetic metal sheets and foils, dipped, plated, or laminated nonmagnetic coatings on ferrous sheets, and ferrous coatings on nonmagnetic sheet materials.

The system affords indication and recording of the thickness of the sheet material being produced. The principal application of this is to gaging of the finished sheet at the dry end of a paper machine. In addition, when used with a controller and a gate regulator, speed regulator, wing regulator, or combination of such regulators, it affords control of the thickness of the sheet being produced, as for example, at the wet end of a paper machine.

### INDICATING AND RECORDING EQUIPMENT

Each unit of the indicating and recording equipment consists essentially of a gaging head with mounting, an amplifier, a gage selector, and an indicating and recording meter. Any

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desired number of units may be utilized for determining the thickness of the sheet at various points across its width. Usually three units are employed at the wet end of a cylinder-type boxboard machine and one or more at the dry end. A cabinet is provided for housing the amplifiers, gage selectors, meters, and the adjunctive indicator lights, control dials, etc.

The gaging head consists of a stainless steel roller mounted to ride on the sheet being measured with provision in the mounting for readily lifting all gaging heads from the sheet when not in use. The gaging roller houses an electromagnet (actually a transformer with one end of the core open). Alternating current (400 cycle) is supplied to one winding of the transformer coil and the current induced in the other coil varies in amplitude with the distance between the open end of the core and a magnetic shunt. The latter is a steel roller (in most cases one of the drier drums of the paper machine or an idler roller). Since the gaging roller rides on the sheet, which is in contact with the reference roller or shunt, the gap between the latter and the open end of the transformer core varies with the thickness of the sheet and, as the sheet thickness varies, the current induced in the secondary transformer coil varies accordingly. This method of gaging obviates the difficulties inherent in mechanical, photoelectric, or other systems of gaging which require a fixed reference point, since, with proper damping of the circuit, it is not adversely affected by vibration, eccentricity in the reference roller and spot, or transitory variations in the sheet thickness which occur too frequently to be controlled.

The amplifier circuit is so designed that the amplitude of the signal supplied through it, from the gaging head and gaging selector, to the meter varies in direct proportion to variations in the air gap, which represents the thickness of the sheet. Since the flux density in the magnetic circuit does not vary uniformly with uniform changes from a narrow gap to a wide gap, the amplifier incorporates an automatic gain control which, together with the gage selector, makes it possible to cover a considerable range of thickness without recalibration. The usual range obtainable is 0 to 150 thousandths of an inch, but this can be increased or sheets of greater thickness can be gaged by shifting the range when the low end of the normal range is not needed. In addition to the automatic gain control and amplifying portions of the amplifier, supply circuits for all special voltages used within the system are incorporated on the same chassis. A separate constant voltage transformer for the 115-v., 60-cycle input current is also furnished.

The gage selector utilizes three transformers substantially identical with that in the gaging head. The value of the signal from the gaging head transformer represents the unknown thickness of the sheet to be determined. The signal from one of the three transformers in the gage selector is of known value, which is adjustable by setting of the gage selector dial. All three transformers in the gage selector are mounted on a common carriage which is moved, by adjustment of the dial, toward and away from a steel plate which acts as a variable shunt for all three transformers. Two of the transformers in the gage selector are called "tolerance" transformers and are set, respectively, with a slightly narrower and a slightly wider spacing from the shunt, as compared with that of the transformer from which the "known" signal is obtained. The four transformers (one in the gaging head and three in the gage selector) are so coupled with each other and with the amplifier that the net signal supplied from the amplifier to the meter varies in direct proportion to the gap at the unknown or gaging head transformer (which represents the thickness of the sheet to be gaged).

The indicating and recording meter has an indicating dial graduated to read in thousandths of an inch and a recording pen operating on a roll-type chart similarly graduated. The meter dial and chart have a zero center and, for wet end equipment, usually read to 5 (representing 0.005 in.) on either side of zero, with numbered main divisions representing 0.001 in.

*J. W. Jelks, The Jelks Co.; C. M. Noble, Electric Eye Equipment Co.; Warren Kumblad, Socony-Vacuum Oil Co.; and S. J. Young, Crown Zellerbach Corp.*

and unnumbered intermediate divisions representing 0.0002 in. The range of the meter scale and chart may be altered to suit requirements and for dry end equipment usually reads only to 2.5 (representing 0.0025 in.) on either side of zero. By setting the gage selector dial at a reading corresponding to the desired thickness of the sheet to be gaged, the meter will read zero when a sheet of this desired thickness is being produced. Deviations of the meter needle and pen to the right of the zero center indicate and record variations of the sheet in a plus direction (i.e., increases in its thickness) from that for which the gage selector dial is set. Deviations to the left of zero indicate and record variations of the sheet in a negative direction (decreases in its thickness) from that for which the gage selector is set.

## STANDARD CONTROLLER

The controller usually supplied as standard equipment for use with wet end indicating and recording equipment on paper machines utilizes a series of timers and relays through which a sequence of steps is accomplished. Completion of the sequence results in a correction being made by the gate regulator, in the amount of stock being supplied to the paper machine for incorporation in the sheet.

Several factors typical in the production of paper and boxboard lend complexity to the design and operation of means for accomplishing automatic caliper and weight control. The normal procedure for altering the thickness of the sheet is to increase or decrease the rate of flow of the stock being fed to the machine. This is usually done at the headbox by regulating the position of gage, under or over which the stock flows to the screen mold (on a cylinder-type machine) or to the wire (on a fourdrinier-type machine). The machine is provided with means (comprising "wings" on a cylinder-type machine) for distributing the stock uniformly across the cylinder to keep the sheet thickness uniform transverse to its direction of travel. With our standard controller, regulation of the thickness across the sheet is left to the machine tender. With gaging heads at three positions across the width of the sheet (front, middle, and back) the corresponding recording meters and indicator lights in the control cabinet tell the machine tender, at a glance, whether the sheet is sufficiently uniform in thickness across its width and, if not, which section or sections require adjustment. Under such conditions the three indicating and recording meters must be in agreement before an automatic correction can be made in the sheet thickness (i.e., the sheet must be either light or heavy at all three gaging positions). Only when this occurs does the standard controller initiate the control cycle.

Another condition characteristic of paper and boxboard machines is the substantial time lag (usually 3 to 4 min.) between the point where the flow of stock can be regulated and the point where the thickness of the sheet can be measured. Obviously, this rules out any attempt to make very frequent

corrections at the headbox gate. In other words, small and transitory variations in the thickness of the sheet, which occur frequently in the production of boxboard, are ignored. With reasonably good stocks and otherwise proper operation of the machine, they are not of sufficient magnitude nor duration to require correction.

The standard controller was designed with the above considerations in mind and, with reasonably good stock consistency, proper drying, proper calendering, etc., this type of control has been adequate for holding boxboard to within approximately plus or minus 0.0005 in. of the selected, finished caliper. To accomplish this, the controller uses three adjustable timers. The first timer to come into play measures an "alerting" period. It starts to mark off the time for which it has been previously set when the tolerance setting is violated in the same direction at all of the gaging positions across the width of the sheet. When, for example, three gaging heads are used, the condition which will initiate the control cycle is indicated at the control cabinet by the simultaneous illumination of the three red or the three green indicator lights. If this condition persists for the time interval set on the alerting timer, the latter will run its course and bring the second timer into operation. This second or "correction" timer transmits an impulse to a contactor which operates the correction motor on the gate regulator, causing the gate to be lifted or lowered for the time interval selected on the correction timer. Once the correction timer is started, it and the remainder of the correction cycle runs its course regardless of whether the three-light condition persists or terminates. However, interruption of the three-light condition while the alerting timer is running will cause it to reset, so that the cycle is broken and must be started by a reoccurrence of the three-light condition before a correction is made.

As soon as a gate correction has been made, the third or "confirming delay" timer starts to operate for its present interval, which is slightly longer than the aforementioned time required for the sheet of altered thickness to reach the gaging position. If, at the end of this confirming delay period, the sheet has returned to a thickness within the established tolerance limits, the correction cycle is complete and is held in readiness for another start. If the correction made was not sufficient to bring the sheet thickness back to within the tolerance limits another correction is made in the same direction and another confirming delay period is initiated, without passing through the alerting period. If too great a correction has been made or if other conditions have meanwhile caused the sheet thickness to vary in the same direction as the correction, such that the sheet thickness is outside the tolerance limits in a direction opposite to that which caused the correction, an opposite correction is made immediately at the end of the confirming delay period. Thus, once the alerting timer has run its course and initiated a correction, that portion of the correction cycle involving operation of the correction timer and the confirming delay timer is repeated as many times as necessary to re-establish the desired sheet thickness.

## GATE REGULATOR

The gate regulator utilizes a back-geared electric motor operated through a reversing contactor and driving a sprocket. A chain passing over the drive sprocket and idler sprockets is coupled to a vertical rod which is connected at its lower end to the gate to be regulated.

Although boxboard is often composed of several different pulp stocks which are built up on the felt by separate cylinders, the bulk of the sheet is usually filler stock. Therefore, in most instances, automatic control of only the filler stock gate will suffice for controlling the sheet thickness. However, when automatic control of any or all of the other stocks, such as top liner, bottom liner, or underliner is desired, separate gate regulators operated through a common controller may be utilized.

## AVERAGING CONTROL

By minor alteration of the circuit and the use of an additional meter, it is possible to effect control of the sheet thickness in response to variations in its average thickness at two or more gaging positions. In doing this, the added averaging meter is connected with the several gaging heads and gage selectors in such a way that it indicates and records the algebraic sum of the readings on the other meters. The plus and minus tolerances to be maintained are set on either side of zero on the averaging meter rather than on the individual gaging position meters.

Installations of this type have been made on cylinder-type boxboard machines and the mill superintendent, electrician, and machine tenders all report, after several months' use, that they definitely prefer it to the former, conventional system. It obviates the necessity of waiting to make a correction until tolerances are violated in the same direction at each of the several gaging positions. By holding better average caliper at the wet end, it affords closer control of the basis weight of the sheet.

## DIRECT READING OR CHART-MARKER RECORDING

To be of greatest service as a record of the caliper of the finished sheet, as at the dry end of a paper machine, it is necessary that the chart indicate not only deviations from the desired caliper but also what the desired caliper is for the particular product represented by that chart. Since the gage selector dial should be set and left undisturbed at a reading corresponding to the desired caliper, periodic recording on the chart of the actual dial setting will serve two purposes. It will show when and to what extent the dial setting of the gage selector is altered. It will show also, by recording where the gage selector dial is set, what the desired caliper is for the sheet being made at the time of the recording.

To accomplish the above, the dry end recording system now sold for most installations incorporates an addition to the gage selector (called the gage setting transmitter), a timer and a chart having two graduated scales in addition to the zero center scale.

In normal operation the meter pen will simply record variations in the caliper of the sheet which should be read from the zero center chart scale. But at intervals determined by the timer utilized (say every 15 min), two resistances, each of a value determined by the setting of the gage selector, will be cut successively into the meter circuit for a brief period in place of the signal from the gaging head. This causes two successive, momentary, deflections of the meter needle and pen, which are read, respectively, from the short-range and the long-range scales on the chart. On the first deflection, the pen will assume a position on the chart representing digits of the gage selector setting. On the second deflection, it will assume a position on the chart representing tens of the gage selector setting. After these deflections have been momentarily recorded, the timer returns the circuit to normal functioning.

I have attempted to outline the various components which go into a complete installation of the Hurletron control. Since this is a technical meeting, the economic and quality factors which influence a decision to purchase such equipment must necessarily take second place. However, I believe your management would tell you unless equipment pays for itself within a reasonable period of time, or if a marked improvement in quality is not noticed immediately, there is relatively little value in further complicating an already complex operation.

It has been our experience that a company with more than one machine has always purchased additional Hurletron installations after the first one has been in operation over a period of time. I think I am safe in assuming that such a decision would never be made if the installations were not proving themselves from an economic and quality standpoint.

Installations have been made on kraft and all types of

board. Currently we are preparing to make an installation on a fourdrinier machine in Michigan. When initial tests have been concluded and actual experience gained on production runs, it will be quite interesting to prepare a report for you as I know many of you are interested in that application of the Hurletron. We had a simple test rig at a small plant close to Danville some time ago, but the operation was not modern enough to evaluate the equipment properly. We believe that the installation now getting under way will be much more representative of the qualities of the Hurletron and will enable us to give you factual information relating to its operation and efficiency

## Developments in Flotation Deinking of Waste Paper

*J. W. Jelks*

TODAY, the paper industry finds itself in somewhat the same position that the copper producing industry and the mining industry, in general, were in near the turn of this century, and we, like the copper producers, can use the froth flotation process to solve a lot of our problems.

The copper producers found themselves with only low-grade ores available. Shipping low-grade ores meant that freight costs would consume the profits. It developed that the froth flotation process made it possible for them to upgrade the low-grade porphyry copper ores and thereby profitably open vast reserves that were heretofore of no economic importance.

While the paper industry is not in the identical position of the copper producers there are, nevertheless, a number of parallels to our problems today. In our case we are faced with rising costs, of being prohibited from discharging large quantities of waste water into the streams, and with the problem of having more difficult materials to remove from one of our low-cost raw materials, waste paper. At the same time we are faced with the demand to produce a better product, and this is where we can profit from the mining industry.

Properly applied the froth flotation process which today is universally used to make mining pay profits, can go a long way toward solving a number of these problems for our industry. (1) It can lower costs by permitting low-grade waste paper to be upgraded and used in the finest of papers. (2) The problem of stream pollution, generally associated with the paper recovery process, is virtually eliminated by use of the froth flotation process. The quantities of waste effluent from the flotation process are very small by comparison to conventional processes involving washing. This, coupled with the exceedingly high yields flotation gives, makes it an effective answer to a number of problems faced by the deinking mill.

The froth flotation process works on the basis of surface differences. Because of different surface characteristics many different materials in a slurry can be separated from one another. This is first accomplished by establishing a stable air bubble, and then attaching a part of the slurry that is to be removed to the air bubble. It could be compared to attaching a specific type of stone from a driveway to a buoyant balloon.

It can thus be seen that the process is not necessarily dependent on the size of the particle, or on the specific gravity of the material to be removed. For instance one of the easiest applications for the froth flotation process is to separate lead sulphide from a slurry of lead sulphide and limestone. Although the lead sulphide is several times as heavy as the limestone it nevertheless floats due to the fact that the buoyant bubbles attach themselves to and lift the heavier material to the top of the slurry in the form of froth.

J. W. JELKS, The Jelks Co., Middletown, Ohio.

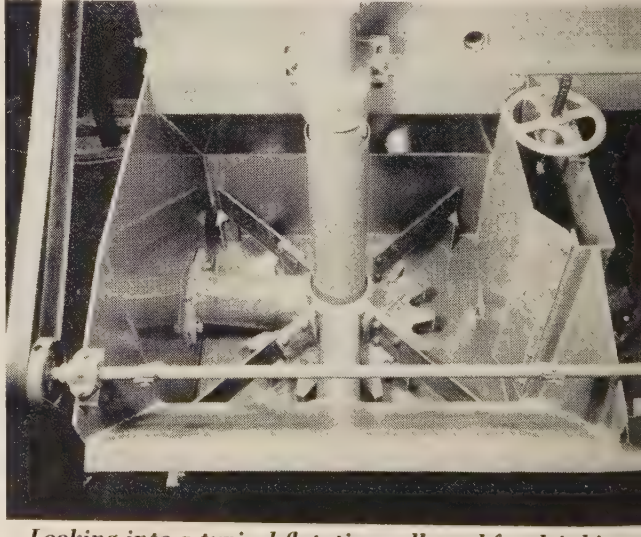
*An 8-cell Denver "Sub-A" paper flotation machine a factory prior to shipment*

The required stable bubble is established by the use of a chemical reagent called a frother. A frother generally consists of a long chain molecule that has one hydroxyl ion attached. It further has to be nearly, but not quite, insoluble and slightly ionizing. This type of a material will form a sort of fuzz around a bubble, or interface, consisting of the long chain portion of the frother molecule. This establishes a like charge around the bubbles and has the effect of making them repel each other thus establishing the required stable bubble.

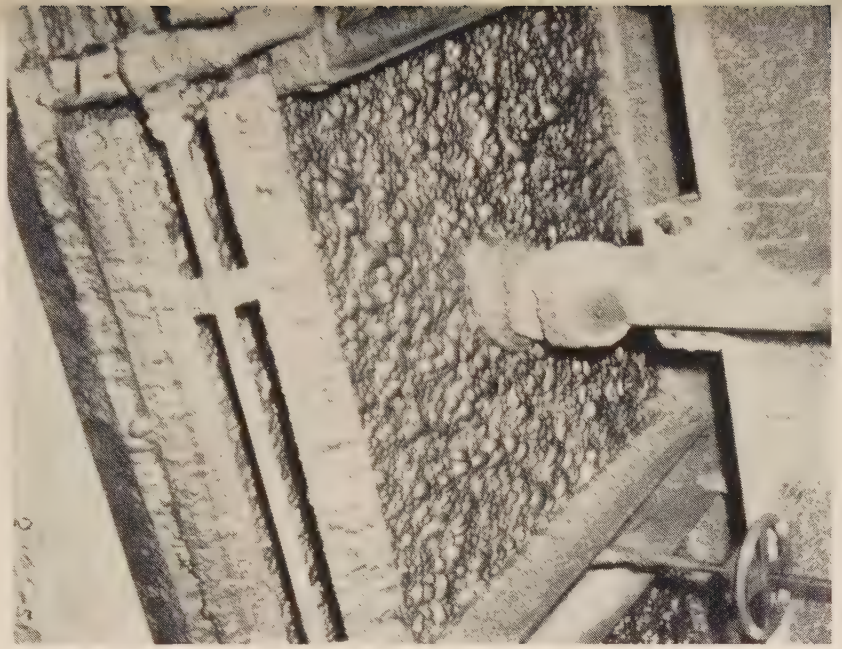
The next thing needed is a collector. The collector must be highly selective and be attracted to the material to be removed from the slurry. Finally, it must have a strong affinity for the interface of the bubbles just discussed. In order to fill this requirement a good collector should be insoluble and should have a surface that repels water. The fatty acids their insoluble soaps, hydrocarbons, and like material are representative of "collector reagents."

To illustrate how these various factors come into play in the deinking of paper by the froth flotation process, pine oil or one of the higher alcohols is generally used as the frothing agent.

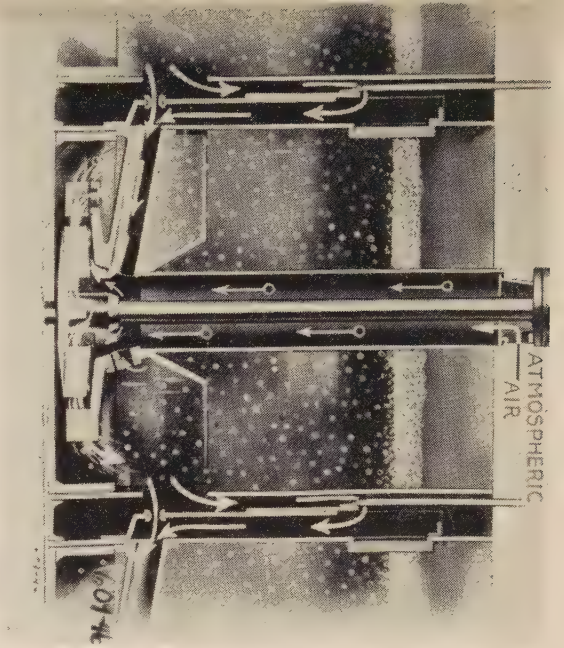
For the "collector" a material such as oleic or stearic acid is often added to the cook, and prior to the flotation step, a heavy metal salt such as zinc sulphate will be added. This results in the precipitation of an insoluble zinc soap of the fatty acid which is a good collector. Generally this collector is assisted by the addition of kerosene which results in further collecting.



*Looking into a typical flotation cell used for deinking*



*View of top of a flotation cell*




***Cut-away drawing showing the mechanism of flotation cell and its operation***

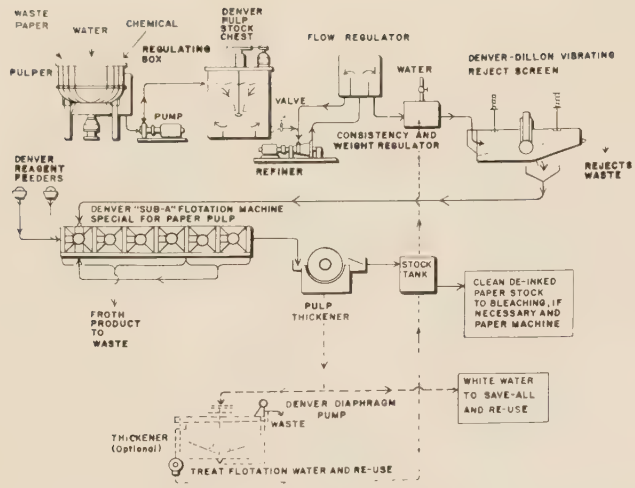
The history of the froth flotation process dates back into antiquity. There is the quotation from Herodotus stating that although he had not personally seen it, he had heard of Byzantian maidens that drew up gold dust by dipping pitch-covered feathers in the mud. The Golden Fleece of mythology probably had its origin from the practice of letting gold-bearing mud flow over a grease soaked sheep's skin that had not been dehaired. Cornelio L. Sagui states that the ancient Greeks used "skin flotation" in recovering the metallic particles in their crushed ore, which, if so, indicates that man has been aware of the forces that make the froth flotation process operate for a long, long time.

Finally from the realm of legend, every mining area has its

**Development of Fixed Costs**

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| I | Financial—It is assumed that a plant can be erected for $175,000 that will produce 30 tons per 24 hrs. | Plant to be written off in 5 yr., 175,000/5, 35,000 | Insurance and interest taken at 7%, 175,000 × 0.07, 12,250 | Amt. reqd. annually 47,250 | 30 × 300 operating days = 9000 tons annually | Cost per ton, 47,250/9000 hr. $5.25 → | $5.50 will be used per ton to allow for some contingency $5.50 per ton |
| II | Labor (direct)—It is estimated that four men per shift can operate the plant | 1—operator<br>1—lap operator<br>1—handling stock<br>4 total | | Average labor cost taken as $1.50 per hr.<br>4 × 8 × 3 × $1.50 = $144, cost per ton, 144/30 = $4.80 | | | 4.80 per ton |
| III | Power | Est. requirement | | Pulping..... $0.60<br>Refining..... 0.60<br>Pulper dump pump... 0.25<br>Pump to float..... 0.10<br>$1.55 | 6 bl. stages pump.... 90.0<br>5 sta. washing..... 37.5<br>Centricleaning..... 40.0<br>Screen..... 7.5<br>175.0 | Flotation..... 80<br>Lap..... 5<br>85<br>155<br>175<br>415 | Assuming 1 hp. = 1 kw. and 1 kw. costs 1¢, $\frac{415 \times 24 \times .01}{30}$ = 3.32 per ton<br>3.32 |
| IV | Water | Est. requirements | | 30,000 gal. at 2½¢ per 1,000 | | | 0.75 per ton |
| V | Bldg. heat | | | | | | 0.24 per ton |
| VI | Maintenance | | | | | | 1.50 per ton |
| | | | | | | | $16.11 per ton |

*Note:* The bleaching costs are based on a new type of equipment and may be low compared to a conventional bleach plant.




***Wastepaper recovery system***

story of the miner's wife that noticed specs of valuable mineral floating on the suds in her wash tub after washing her husband's dirty and greasy cloths.

Record has a somewhat different story to tell of the development than the miner's wife. The secrets of the mechanism of the froth flotation process have been obtained in steps. Haynes, around 1860, seems to have noted first the differences of wettability of various minerals. From this beginning the so-called "bulk oil flotation process" evolved. This consisted of mixing a large quantity of suitable oil with the mineral and allowing the oil to pick up the values while being agitated with the ground ore, after which the oil was allowed to float to the surface where it was collected and the mineral removed. Harry Fischer of Lockland, Ohio, was granted a patent for deinking paper that closely resembles this process. It is interesting to note that the oils used in the bulk oil flotation processes are for the most part used as collectors in modern practice.

Skin flotation was the next step and generally consisted of establishing a skin of oil on top of the ore slurry and moving the slurry so as to come in contact with the skin of oil. The purpose of this development was to lower the amount of oil required.

Alcide Froment can probably be considered the father of froth flotation as he obtained the first patent, a British patent, using a gas bubble as the buoyant medium. His patent noted that gas liberated from an acid solution in which there was a sulphide or carbonate present would pick up particles of sulphide minerals. However it was for Sulman, Picard, and Ballot to note that air would serve the purpose of providing the buoyant medium thus evolving the froth flotation process to a point essentially as we know it today.

Pierre Hines of Portland, Ore., seems to have been the first to recognize the possibilities of applying the process to deinking of paper. He did a thorough job investigating the process for use in the paper industry with the flotation materials available to him in the mid thirties.

The author had the pleasure of starting the first commercial deinking installation using the froth flotation process in 1950. Denver "Sub-A" flotation machines were used and to our knowledge this was the first departure from the conventional washing methods that have been in use since paper was first deinked around 1730.

Since the first commercial installation using the froth-flotation process was installed several other very successful applications have been made and it has been found that the process can potentially solve many other problems that face the paper and pulp manufacturers. These take the form of removing pitch, asphalt, plastic, waste, and a number of other materials in addition to ink removal.

**Costs of Various Grades Plus Cost of Upgrading, Value of Finished Pulp, and Profit**

| | *Grade* | *Cost of stock (delivered)* | *Cost of cook chemicals* | *Cost of flotation chemical* | *Type of bleach and cost* | *Steam cost* | *Gross cost* | *Yield, %* | *Net cost raw material* | *Fixed cost* | *Total cost per finished ton* | *Value of product* | *Profit* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| I | New 100% kraft corrugated cuts | $56.00 | ... | $1.00 | II, $17.57 | $5.68 | $80.25 | 80 | $100.31 | $16.11 | $116.42 | 85% bleached kraft pulp | $ 2.58 |
| II | All kraft, asphalt-free, old corrugated | 45.00 | ... | 1.00 | II, 17.57 | 5.68 | 69.25 | 80 | 86.56 | 16.11 | 102.67 | 140 × 0.85 = $119.00 | 16.33 |
| III | No. 1 sorted brown kraft | 35.00 | ... | 1.00 | I, 11.75 | 5.68 | 53.43 | 85 | 62.86 | 16.11 | 78.97 | 90% bleached kraft pulp | 47.03 |
| IV | New brown kraft bag waste | 70.00 | ... | 1.00 | I, 11.75 | 5.68 | 88.43 | 85 | 104.04 | 16.11 | 120.15 | 140 × 0.9 = 126.00 | 5.85 |
| V | Colored tab cards | 40.00 | ... | 2.34 | I, 11.75 | 5.68 | 59.77 | 85 | 70.32 | 16.11 | 86.43 | 90% bleached kraft and sulphite | 37.32 |
| VI | Manilla tab cards | 55.00 | ... | 2.34 | I, 11.75 | 5.68 | 74.77 | 85 | 87.97 | 16.11 | 104.08 | [(140 + 135)/2] × 0.9 = 123.75 | 19.67 |
| VII | No. 1 colored ledger | 45.00 | $2.04 | 2.34 | III, 6.89 | ... | 56.27 | 90 | 62.52 | 16.11 | 78.63 | 90% bleached sulphite | 42.87 |
| VIII | White ledger | 60.00 | 2.04 | 2.34 | III, 6.89 | ... | 71.27 | 90 | 79.19 | 16.11 | 95.30 | 135 × 0.9 = 121.50 | 26.20 |
| IX | Mixed books and magazines | 25.00 | 1.28 | 2.34 | III, 6.89 | ... | 35.51 | 65 | 54.63 | 16.11 | 70.74 | 90% bleached soda | 55.26 |
| X | No. 1 repacked heavy books and magazines | 40.00 | 1.28 | 2.34 | III, 6.89 | ... | 50.51 | 65 | 77.71 | 16.11 | 93.82 | 140.00 × 0.9 = 126.00 | 32.18 |
| XI | No. 1 news | 18.00 | 1.79 | 2.34 | ... | ... | 22.13 | 95 | 23.30 | 16.11 | 39.41 | 90% groundwood pulp 80.00 × 0.9 = 72.00 | 32.59 |

**Development of Process Costs**

| Type of process | Chemical | Weight per ton reqd., lb. | Cost per pound chemical cents | Chemical cost per ton |
|---|---|---|---|---|
| I Light multistage bleach | 9% chlorine | 180 | 3.83 | $ 6.89 |
| | 9% caustic soda | 180 | 2.55 | 4.59 |
| | 1.5% sulphur dioxide | 30 | 0.90 | 0.27 |
| | | | | $11.75 |
| II Heavy multistage bleach | 15% chlorine | 300 | 3.83 | $11.49 |
| | 11.4% caustic soda | 228 | 2.55 | 5.81 |
| | 1.5% sulphur dioxide | 30 | 0.90 | 0.27 |
| | | | | $17.57 |
| III Single stage hypo bleach | 5% chlorine | 100 | 3.83 | $ 3.83 |
| | 6% caustic soda | 120 | 2.55 | 3.06 |
| | | | | $ 6.89 |
| IV Cook chemicals for unfilled chemical pulp stock (ledger, etc.) | 4% caustic soda | 80 | 2.55 | $ 2.04 |
| V Cook chemicals for filled chemical pulp stocks (magazine, books, etc.) | 2.5% caustic soda | 50 | 2.55 | $ 1.28 |
| VI Cook chemicals for unfilled groundwood stocks (news, etc.) | 0.5% caustic soda | 10 | 2.55 | $ 0.26 |
| | 1% soda ash | 20 | 1.65 | 0.33 |
| | 4 lb. wetting agent | 4 | 30.00 | 1.20 |
| | | | | $ 1.79 |
| VI Flotation chemicals for heavy circuit | Zinc sulphate | 7.5 | 9.5 | $ 0.71 |
| | Pine oil | 5 | 14.0 | 0.70 |
| | Alcohol modifier | 1 | 25.0 | 0.25 |
| | Kerosene | 5 gal. | 13.5 per gal. | 0.68 |
| | | | | $ 2.34 |
| VII Flotation chemicals for light circuit | Pine oil | 3 | 14.0 | $ 0.42 |
| | Fatty acid | 1 | 24.0 | 0.24 |
| | Kerosene | 2.5 gal. | 13.5 | 0.34 |
| | | | | $ 1.00 |

*Steam required for caustic extraction*

A. 1. It is assumed that a 5.5% consistency will be used so as to allow use of low density pulp.
2. 50°F. inlet temperature is assumed. 180°F. temperature at which extraction stage will be operated.

B. For each pound of stock at 5.5% consistency it will be noted that 17.18 lb. $H_2O$ will be presented.

C. Heat reqd.:
1. For water, 17.18 × 2000 (180 − 50) = 34,360 (130) = 4,466,800
2. For pulp, 2000 × 0.4 × 130 = 104,000
Total B.t.u. 4,570,800

D. Heat in steam:
1. Total heat at 150 p.s.i.g. 1195 B.t.u. lb.
Heat in 180° $H_2O$ 148 B.t.u. lb.
2. Heat available in 1 lb. steam 1047 B.t.u. lb.

E. Steam reqd. per extraction stage: 4,470,800/1047 = 4366 lb.

F. Cost, assuming 65¢ per 1000 lb.: 4.366 × 0.65 = $2.84 per ton.

Two stage, 5.68

To date. the froth flotation process has been most successfully applied to the deinking of unfilled grades of waste paper, such as newsprint, ledger, and the like.

One installation here in New York State is using Denver "Sub-A" flotation machines deinking newsprint and is supplying pulp that was formerly obtained by grinding wood. This installation gives yields of 95 to 96% of a pulp that is about 50 G.E. brightness. The chemical cost per ton is $4.85* and the water required per ton is 900 to 1100 gal. per ton. The stock can be bleached as much as 11 points G.E. brightness by using zinc hydrosulphite and probably by a considerably greater amount by using the peroxide method, although this remains to be seen.

In another installation part of the stock is subjected to the froth flotation process. This is done to reduce the amount of water required by this deinking plant that is processing ledger. A partial flotation effect is obtained in a settling trough in that the larger particles of ink come to the surface of the settling trough. In this installation the upper portion of the stock is taken from the settling trough and run through a Denver "Sub-A" flotation machine giving a proportional water saving. This permits the use of a minimum size installation that provides an efficient removal of the larger ink particles.

Magazines and other filled stocks have been deinked by the froth flotation process. While other applications have been successful to a point, there nevertheless is room for considerable improvement. On a laboratory scale we have been able to achieve the needed improvements and hope to be able to have commercial installations using the improved techniques on certain difficult problems in operation in the not too distant future.

The great drawback in deinking filled stocks is that a finely divided ink will adsorb on the surface of the fillers present. As it is desirable to retain these fillers this adsorption prevents adequate separation and thus cancels some of the advantage to be had from the froth flotation process. We have found that proper preparation of the stock prior to flotation can completely solve this problem and provide a white filler. By going back into mining and ore dressing practice it is noted that a very definite importance is placed on the method in which the ore is ground or pulverized to liberate minute particles of mineral. This step in mining is the exact parallel of the cooking and pulping steps in the deinking of paper. As our problem is to liberate or get the ink off the paper and out of the slurry we immediately find a proper size particle is necessary to effect an ideal flotation condition.

If the ink finish is removed from the paper and broken only sufficiently to get it off the paper there will be no adsorption by the filler as the very fine particles necessary to adsorb will not be liberated. This can be accomplished by first soaking the waste paper in a detergent solution sufficiently long enough to break the bond between the paper and ink. It has been found that a whole magazine can be so treated. If the paper so treated is pulped sufficiently to be pumped and immediately floated it is found that a sheet of 63 to 66 G.E. brightness before bleaching can be produced easily using a magazine finish. It is further noted that this sheet can be easily bleached to 75 to 80 G.E. brightness, depending on the furnish in the original sheet. On the other hand, it is difficult to get a sheet of 60 G.E. brightness when conventional cooking and pulping methods are used prior to flotation. This is, no doubt, due to the fact that the fine pigment particles necessary for adsorption have not been liberated and the clay has thus not darkened due to adsorption.

The additional step necessary to accomplish this outstanding performance is more than justified as 70 to 80% of the original cooking chemicals can be recovered and used on the next batch coming through which results in a proportional savings in chemical costs.

Laboratory results indicate that the froth flotation process can be applied to a number of other paper and pulp problems. A sample of bleached aspen semichemical pulp was submitted that had in it a number of "pepper spots" that were said to be pitch. This sample was effected with ease, in fact this stock was one of the easiest and required less flotation time of any run to date. This indicates that the process can quite possibly be used for pitch removal from virgin pulps.

Considerable work has been done on the removal of asphalt and it has been noted that this is a very easy material to remove from a bleached pulp. Unfortunately to date, it is difficult to get as clean a separation from brown stocks as

* This company has $4.13 in work sheets as they use a little $H_2O_2$ we do not recommend.

from bleached stocks. The brown stock tends to float along with the asphalt which probably indicates that stocks of this type have some sort of collector such as rosin, tall oil, or like material on the surface which interferes with the flotation. The bleaching process probably removes these materials.

Rubber and plastic materials have been investigated and the froth flotation process shows promise of being able to remove a number of these materials. The surfaces of some of these materials are such as to facilitate their removal with ease while others require that their surfaces be altered for best results.

Raw rubber such as is introduced from the pressure-sensitive tapes is a good example of the latter. This raw rubber wets easily and is therefore not floated. Keeping in mind that the surface is an important thing it is noted that there are a number of reactions that will alter this surface and provide the differentiation necessary to remove the material, the simplest reaction being the action of the halogens on rubber. If the stock is subjected to bleaching, the chlorine will react sufficiently to alter the surface sufficiently to obtain a separation. Other reactions are the reaction of raw rubber with sulphur, selenium, or the peroxides of various organic materials. It should be pointed out that this gives a complete field in which to work that can provide an endless number of successful separations. One other point that should be brought up is that the rubber will not respond nearly so well if the original cooking or detergent solution remains in the circuit as it has a decidedly depressant effect.

Another class of materials is being investigated, namely the removal of objectionable synthetic fibers from rag stock. These are nylon, rayon, etc., which make an objectionable spot on the sheet as they are thermoplastic and melt when the sheet is passed over the drier. To date, this work has not progressed to a point of having established the definite boundary of its possibility although results look very promising.

In conclusion, it can be noted that any time the problem arises that involves an undesirable particle in paper stock, the froth flotation process can possibly solve the problem. When less water usage is indicated in a washing problem the froth flotation process again holds great promise of solving this, too, as water is a working medium in this process rather than an active participant as in the washing processes. Thus by its full utilization the froth flotation process can to a great extent solve the stream pollution problem as well as help the mill cut its costs and improve its quality.

## Petroleum Waxes and Additives for Paper

*W. S. Kumblad*

Papers dealing with the application of modified plastic films in which synthetic resins constitute the principal ingredient have already been presented at this meeting. The subject which we will discuss today deals with two products of the petroleum industry: refined paraffin and microcrystalline wax. In this instance, waxes will be considered as the principal component in paper coatings and will, for specialty applications, be modified with synthetic resins. We hope that the subject will be of interest, particularly since a major portion of the total production of waxes of both types finds its way into the paper and paper converting industries. Incidentally, recent figures show the annual consumption of waxes by these industries to be approximately 750 million lb. You perhaps will agree that this is a sizable requirement. Accounting for much of this increase in wax consumption is the current popularity of paper milk containers. The following figures show the increase in the ratio of paper to glass containers which has taken place during the past 8 years:

| | Paper | | Glass |
|---|---|---|---|
| 1936 | 0.2 | to | 1 |
| 1942 | 3.0 | to | 1 |
| 1954 | 19.0 | to | 1 |

There are many types of paper packages employing wax. The package which was used for field rations by the armed forces consisted of a paperboard box coated with wax. The paper milk bottle is a somewhat similar container but is waxed both inside and out. Then there are those containers made of laminated papers, either of the fiber-can type or the folding box type. Frozen foods and other products are packaged in liners in a paperboard carton or in a bag-in-box assembly. Materials such as coffee are now sold in multi-ply, heat-sealed paper bags. Laminated papers are also used in packing potato chips, tea, dehydrated soaps and have even, in some cases, replaced the foil wrapper for cigarettes. Waxed single sheets are employed as bread wrappers, butcher papers, locker papers, drinking cup stock, sandwich bags, and in general use around the kitchen. Among the most important elements of a package or a wrapper of these types is the water vapor or greaseproof barrier which it contains. Many materials might serve for this purpose and a large number have been employed at one time or another. However, petroleum waxes are the most widely used and, from the viewpoint of price, supply, and ease of handling, represent the most satisfactory materials for the purpose.

The terms "waterproofness," "moistureproofness," and "water vaporproofness" are frequently loosely used, and as a result some confusion exists regarding their exact meaning. Waterproofness refers to the ability to withstand the passage of liquid water. Water vaporproofness is used to refer to the ability of a barrier to resist the passage of a water vapor, while moistureproofness is used as a general term to indicate resistance to transmission of either liquid water or water vapor. In order to illustrate the difference between the two terms, waterproof and water vaporproof, one may use the desert water bag as an example. This bag depends, for its success, on the fact that it is waterproof but at the same time is capable of transmitting water vapor quite easily. The bag is made of a canvaslike cloth woven so tightly that liquid water cannot pass through it. Water vapor, on the other hand, passes easily through the cloth and evaporates freely into the atmosphere thus cooling the bag and keeping the contents at a relatively low temperature. The cotton or cellulose in the bag can impart no protection against water vapor. In order to produce a completely water vaporproof bag it would be necessary to put a continuous water vapor resisting barrier on the container. This can be done with waxes.

In the paper industry two general types of wax coatings are applied. They are termed "wet-wax" and "dry-wax" coatings. The wet-wax sheet carries substantially all the wax as a surface film with only enough wax impregnated into the paper to effect a bond with it. The dry-wax paper has the wax driven almost entirely into the paper with little if any remaining as a surface film. Dry waxed paper is characterized by water-repellent properties but possesses only limited water vaporproofness. Wet waxed paper, on the other hand, is both waterproof and water vaporproof.

Another type of paper, having the advantages of both wet and dry waxed papers, is laminated paper. In laminated sheets two layers of paper are bonded together with a layer of wax. Generally one of the papers used is relatively smooth, a glassine or a foil, and the second may be less highly finished. Usually the molten wax is applied to the smoother sheet which is then bonded to the more open sheet by pressure. Thus a continuous wax barrier is applied between the sheets and the resulting laminated paper is highly resistant to water vapor. Since the wax film or barrier is protected by paper on each side the continuity and effectiveness of the film are protected against injury which might result from careless handling.

W. S. Kumblad, Staff Engineer, Socony-Vacuum Oil Co., Inc., New York, N. Y.

Barriers have other purposes than preventing the passage of moisture vapor. They impart also greaseproofness, a term used by the industry to indicate resistance of a barrier to the passage of grease. However, since oils and fats are similar in their method of transmission, greaseproofness implies resistance to these materials as well. Greaseproofness is different than either waterproofness or water vaporproofness. While these may be obtained simultaneously, they should not be confused one with the other. It is known that water vapor is transmitted through a sheet of paper by passage along the cellulose fibers and that the course can be arrested by placing a continuous moisture-resistant film on the surface of the paper. Grease and oil, on the other hand, are not carried along the cellulose fibers, which are naturally quite grease resistant, but pass between the fibers through the minute openings left during the formation of the sheet. If the paper is impregnated or coated with wax a barrier is formed to prevent the passage of grease or oil. However, the effectiveness of this barrier is dependent entirely upon how long it resists the solvent action of the oil or grease. This resistance to oil and grease is dependent upon (1) the composition of the wax barrier, (2) time of contact, (3) the thickness of the wax coating, and (4) the temperature of the sheet. Waxes are soluble in oils, fats, and greases to a measurable extent, but solubility varies directly with the melting point of the wax. As the melting point increases, the solubility decreases but at a more rapid rate. For example, a wax melting at 165°F. is only $^1/_{20}$ as soluble as one melting at 133°F. and only $^1/_{40}$ as soluble as one melting at 125°F. The temperature and time of contact are factors in determining greaseproofness because they affect the quantity of wax dissolved in the grease or oil, and the thickness of the wax barrier is a factor because it determines how rapidly the grease will be able to remove the wax and form an opening through the sheet.

Two types of petroleum waxes are available for all these operations—paraffin wax and microcrystalline wax. Fully refined paraffin waxes are relatively hard, brittle, white crystalline solids. They are sold in various melting point grades ranging from about 120 to 150°F. They are desirable as coating materials chiefly because of their hard, dry nature. However, for many applications they are unsuitable because of their brittleness and because of their low melting points. Brittleness is most pronounced at low temperatures and makes the wax vulnerable to rough handling. Refined wax is used for coating paper milk bottle stock and as a component in bread wrap coatings and similar packaging materials.

Microcrystalline waxes are materials obtained by de-oiling crude petrolatum. They are sometimes known as amorphous waxes or petrolatum waxes all of which terms are synonymous. Microcrystalline waxes differ from paraffin waxes in several important respects. They are made up of considerably smaller crystals, hence, the name microcrystalline. They are higher in melting point (145 to 190°F.). They have greater ductility at all temperatures. They are more tacky. They are darker in color. Microcrystalline waxes owe their usefulness to their greater strength, ductility, and their higher melting point. The latter characteristic makes them much more resistant to grease than are paraffin waxes.

The two types of waxes, microcrystalline and paraffin, are often used together in a blend and the following two examples illustrate the effectiveness of microcrystalline wax when employed as a modifying agent in refined paraffin wax. A converter of glassine found that the base sheet, as delivered from the paper machine, contained a number of hard spots, scattered throughout the web. When the glassine was wax coated these spots, present as points of high pressure, caused sticking within the wind-up reel. Upon rewinding, blocking occurred, with small areas of wax actually being pulled away from the base sheet, leaving undesirable depressions or pock marks. The addition of 15% microcrystalline wax to the paraffin completely eliminated the blocking and, in addition, prevented beads of wax from building up on the chill rolls.

***Officers of the Empire State Section. Front: H. A. Spencer, Knowlton Bros.; G. K. Storin, Niagara Alkali Co.; J. D. Parmele, St. Regis Paper Co.; and R. W. Kumler. Standing: R. N. Prince, Eastern Chemicals, Inc.; G. B. Martin, F. C. Huyck & Sons; G. G. Cole, Diamond Match Co.; C. J. Sibler, West Virginia Pulp & Paper Co.; D. C. Mather, Carthage Paper Makers, Inc.; S. J. Young, Crown Zellerbach Corp.; J. J. Forsythe, International Paper Co.; P. L. Haggerty, Geo. La Monte & Son; and R. M. Drummond, International Paper Co.***

In another instance, fish box blanks after waxing with straight paraffin, showed a tendency to block when stacked in piles prior to shipment. The addition of 10% microcrystalline wax not only eliminated the difficulty but also improved the "foldability," along score lines, of the coated blanks.

So much for the use of straight paraffin or microcrystalline waxes, or blends of the two. Now for the use of additives and a brief review of the literature pertaining to the subject. Examination of brochures and articles in the literature and of patents granted indicates that there are three major additives which modify the characteristics of petroleum waxes sufficiently to be of interest to wax consumers now. These are (1) butyl rubber, (2) polyethylene, and (3) polyisobutylene. In addition, there are several others which may be of value in the future, such as polyamides, polyamines, polyisobutylene plus styrene, polyisoprene plus styrene, polyketones, polyvinyl esters, rubber, synthetic alcohols, terpenes, terphenyls, and waxes other then petroleum waxes. We give below the information obtained in the literature survey regarding the effect of a number of these materials on various characteristics of paraffin waxes. Similar effects can be expected with microcrystalline waxes. It should be pointed out that since much of the data given here are from patent literature, they should not be considered as recommendations concerning their use.

*Butyl Rubber.* It is indicated that the addition of a small amount of butyl rubber to paraffin wax increases the tensile strength of the blend until a maximum value is reached at 1.0 to 2.5% butyl rubber. The effect of butyl rubber in quantities above 5% was not given in the literature. Above 1.0% butyl rubber, as the per cent of butyl rubber increases up to 5%, the penetration of a butyl rubber-paraffin wax blend increases. No data were given above 5%. The viscosity of butyl rubber-paraffin wax blends increases as the per cent of butyl rubber increases. The addition of up to 5% butyl rubber (molecular weight 40,000 to 70,000) to paraffin wax (melting point 133/135°F.) has little effect on the melting point of the wax.

*Polyethylene.* The addition of polyethylene in quantities up to 5% increases the tensile strength of the wax. In the range of 0 to 5% the addition of polyethylene to paraffin wax has little effect upon the melting point. Up to 1% of polyethylene in paraffin wax increases the blocking temperatures. A 50-50 blend of polyethylene and polyisobutylene also increases the blocking temperature.

*Polyisobutylene.* The addition of polyisobutylene increases the tensile strength and viscosity of paraffin wax and decreases the blocking temperatures and moisture vapor transmission. It is to be noted that polyisobutylene alone, or in a 50-50 blend with polyethylene, is more effective than polyethylene alone in its ability to increase the tensile strength of wax The addition of as little as 1.25% of polyisobutylene increases the laminating strength of paraffin wax.

*Polyamides.* Polyamides, such as polyhydroxamides, blended with low-melting point paraffin waxes increase the melting point of the blend.

*Copolymer of Polyisobutylene and Styrene.* The addition of up to 1.25% of the copolymer increases the tensile strength of the wax. The laminating strength is increased by adding up to 2.5% of the copolymer. The copolymer of polyisobutylene and styrene increases the viscosity of the wax. Up to 1.25% of the copolymer has little effect on the melting point and penetration, but the effects of higher concentrations were not presented in the literature.

*Polyketones.* Polyketones will increase the tensile strength and hardness of the paraffin wax.

*Synthetic Alcohol Waxes.* These materials appear to increase the solidification point and the viscosity of paraffin waxes.

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# SHIBLEY AWARD PAPERS—1954

*Pacific Section, Longview, Wash., March 19, 1954*

## The "80-Grit-Touch-Up" Method for Maintaining Calibration of the Valley Laboratory Beater

FRANK DINGER

In my experience the question most often asked when reviewing pulp quality results from laboratory beater testing has been: "Are the beaters in calibration?" Because this has been my personal responsibility, my answer has been, "I think so, but I will check the beaters to make certain."

The problems arising from this question are of daily concern at all pulp and paper mills, because quality is the criterion of all finished products. Therefore, a need exists for a uniform and reliable method for conditioning the Valley laboratory beaters and what I am reporting on is the result of my investigations on this problem.

TAPPI Standard T-200 for conditioning the laboratory beater is time consuming and does not appear to give uniform nor reproducible results. Therefore, my problem was to find a method which would possibly give more reliable results.

Several factors which are common to all methods for calibrating the Valley laboratory beaters were studied. These factors were: (1) placement of bedplate and shape of the lead grout, (2) various degrees of surface roughness on the roll bars and bedplate, (3) various degrees of roundness on the roll bar's leading edges, (4) the effect of contact pressure on the beating surfaces when varied by changing the weight on the tackle arm, and (5) mechanical wear of the bars and bedplate. Also, special attention was given to such variables as individual tester's techniques, calibration of other testing equipment, water temperature control, and humidity room conditions.

### EQUIPMENT AND TESTING

The TAPPI standard methods for testing wood pulp for physical properties are used in our laboratory. The equipment consists of the $1^1/_2$-lb. Niagara-type beater, Canadian Standard freeness tester, British Standard sheet mold, Mullen tester (Model C), Elmendorf tear tester, and M.I.T. fold machine. The test sheets are conditioned and tested in the standard humidity room conditions of 73 ± 3°F. and 50 ± 2% R.H.

A standard pulp for checking of beater calibration was selected from a well-aged bale of average strength bleached sulphite pulp. A calibration test was run on the standard pulp in each beater every fifth day. The calibration test was a complete beater test consisting of freeness, consistency, basis weight, bursting strength, tear, fold, and density at regular beating-time intervals.

### PRELIMINARY WORK

I was to start this investigation by making a study of the smooth bar method of calibration which was to be compared with the rough bar method (TAPPI Standard T 200). To obtain the smooth-bar surfaces the following grinding and polishing materials were needed: 80, 120, 220, and 400-grit carborundum of the silicon carbide type and diatomaceous earth.

The grinding in of the bars and bedplate for the smooth-bar method was performed in the roll section of the beater. This section was dammed from the other part of the beater by placing a removable dam just ahead of the roll and another dam between the top of the back fall and beater hood. These dams were made of a stiff material such as sheet metal, $^1/_4$-in. plywood, or $^1/_4$-in. hard rubber. The dams are fitted into place as snuggly as possible, then a water-tight seal was obtained by using an electrician's Duct-Seal putty. This putty was easily handled and had sufficient adhesive power to remain in place, yet it was easily removed without creating an oily or messy condition in the beater.

A sturdy adjustable clamp for clamping the bedplate lever arm during the grinding in steps was made available.

Each piece of testing equipment that was to be used during the study was carefully checked, and all necessary repairs or replacements were made. The beaters were repaired to the extent of new bars, bedplate, bearings, and rubber diaphragm.

### PROCEDURE AND RESULTS

One beater was selected for the TAPPI Standard method of calibration and another beater for the study of other methods of calibration.

The placement of the bedplate in these two beaters was given utmost care so as to obtain the optimum placement as shown in Fig. 1. A properly placed bedplate and a correctly shaped lead grout were necessary to obtain satisfactory beater operation. The bedplate must be set directly under the center of the roll and at an elevation such that when in contact with the roll bars the top edge of the bedplate holder will be in alignment with the bottom of the tub. The lead grout which retains the bedplate in the bedplate holder should be built up and shaped to conform with the bottom edges of the beater tub. It should be further pointed out that a gentle slope of the retaining lead grout ahead of the roll was important. A very flat or steep slope at this point must be avoided. This can be accomplished by shimming up the bedplate before grouting in. In a similar manner the lead at the back edge of the holder must be built up to conform with the bottom edge of the backfall.

The beater selected for the TAPPI Standard method of calibration was ground in and conditioned as outlined in TAPPI Standard method T 200. The other beater was conditioned for the smooth-bar calibration as follows.

#### *Grinding*

*120-Grit Carborundum.* The roll section of the beater was dammed as outlined earlier. Two hundred grams of 120-grit carborundum were added to a sufficient quantity of water in the dammed section of the beater to provide good circulation. While the abrasive mixture was circulating in the dammed section of the beater the bedplate was brought into contact with the roll bars. The contact pressure was adjusted with 5 kg. on the bedplate lever arm. With this pressure on the surfaces being ground in, the arm was clamped into place.

Frank Dinger, Puget Sound Pulp & Timber Co., Bellingham, Wash.

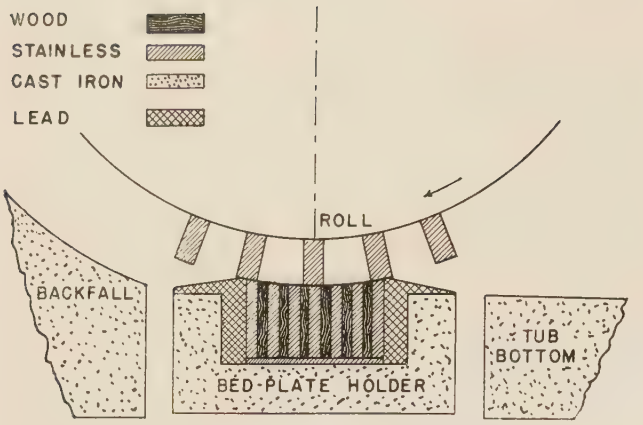


*Fig. 1. Optimum placement of bedplate*

Throughout the grinding period the contact pressure was maintained by adjusting the arm clamp, and it was found that 5 to 15 min. were required to grind the surfaces. At the completion of this grinding step a uniform grating sound will be transmitted through the lever arm, and a uniform gray color will appear on the bar surfaces.

A final check as to the completeness of the grinding was determined from an impression of the bedplate. The impression was obtained by placing two sheets of paper separated by a sheet of carbon paper between the roll and the bedplate. The bedplate was engaged with the roll and while firmly holding the paper the roll was turned about one-half turn. A complete grind will produce an impression showing all seven bedplate bars.

On completing the first grinding step the burs were removed from the roll bars with a file or emery stone and the beater was thoroughly washed out so as to remove all traces of metal and abrasive.

*Important,* the bedplate lever arm must be clamped in place throughout this and all subsequent grinding steps. The roll would be ground out of round if this procedure was not followed. Chattering indicates too much pressure on the surface being ground.

*220-Grit Carborundum.* The dams were replaced for the second grinding step, and 200 grams of 220-grit carborundum were added to the water in the dammed section. The same procedure was followed in this step as outlined in the previous step. The grinding was continued until a uniform gray surface appears on the bars. Again, the burs were removed from the bars, and the beater was washed free of all traces of metal and abrasive. The dams were replaced for the third grinding step.

*400-Grit Carborundum.* Three hundred grams of 400-grit carborundum were added to the water in the dammed section for the final grinding step.

Caution must be exercised throughout this grinding step because with the fine abrasive material the contact pressure should at no time be great enough to bring the metal surfaces of the bars and bedplate into contact with each other. The bar and bedplate surfaces will seize and bur if contact is made. The proper grinding pressure can be judged from the high-pitched grating sound which will be transmitted through the bedplate lever arm. A very fine gray-colored surface on the bars indicates completion of this grinding step.

The burs were again removed from the bars and the leading edge of each bar was rounded to approximately $^1/_{32}$ in. radius. A very fine file or a fine stone was found to be a very convenient tool for rounding the leading edges. The beater was again thoroughly cleaned of all metal and abrasive materials, before proceeding with the polishing steps.

### *Polishing*

The weight on the bedplate lever arm was adjusted to 5.0 kg. The beater was furnished with a charge of standard bleached pulp. This pulp was beaten (about 4 hrs.) to a very low freeness. Then 100 grams of diatomaceous earth were added to the well-beaten pulp, and the beating was continued for an additional 2 hr. A second charge of bleached pulp was furnished to the beater which was beaten for 2 hr.

Following these two polishing steps the beater was checked for beating time with the standard calibration pulp. The beating time to 500 ml. Schopper-Riegler or 250 ml. Canadian Standard freeness was 60 ± 5 min. The beating time to the desired freeness was adjusted by changing the bedplate lever-arm weight. The properly adjusted beater was then checked for strength development with the standard calibration pulp.

1. Normal strength and a fast beating rate would indicate too much weight on the arm.
2. Low strength usually indicates too much cutting. Thus, further rounding of the leading edges may be required.

The above outlined procedure for grinding and polishing the beating surfaces was very carefully followed. The test results from one of several series are shown in Table I. Each series of attempts and adjustments failed to give uniform or comparable results as obtained with the TAPPI conditioned beater.

**Table I. Smooth Bar Beater Calibration Test Results**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* |
|---|---|---|---|
| X-19 | 39 | 102 | 3.0 |
| X-20 | 58 | 104 | 3.0 |
| X-21 | 59 | 104 | 5.0 |
| X-22 | 56 | 104 | 6.0 |
| X-23 | 130 | 106 | 7.0 |
| Tappi $\bar{X}$ | 43 | 114 | 5.5 |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight on tackle arm in kilograms. TAPPI $\bar{X}$ = average of test in TAPPI calibrated beater. Pulp = standard calibration pulp.

The values shown in Table I are for beating time and bursting strength at 250 ml. Canadian Standard freeness. The tests run in the smooth-bar beater are represented by test no. X-19 to X-23 and for comparison TAPPI X represents the average value of five calibration tests on the same standard pulp in the beater calibrated by the TAPPI Standard method.

The first test of the series (X-19) developed 12 points of burst below the TAPPI $\bar{X}$ value. The low strength and the fast beating time seemed to indicate excessive cutting. Thus the bars' leading edges were further rounded before running test X-20 and again before running test X-21. The beating time increased significantly but no change in burst development was obtained. Test X-22 was run following additional polishing with bleached pulp and diatomaceous earth. Again no significant change in bursting strength development was produced.

The next calibration test (X-23) of this series was run after cutting away and rounding the retaining lead at the point of contact with the roll bar. With the retaining lead in this condition the test results did not produce a significant change in bursting strength. The very long beating time would indicate a minimum of fiber cutting was taking place.

Each time the beater was reground and repolished by this method the calibration test results were similar to those presented in Table I. Since the strength development continued at a low level, I decided to substitute other polishing materials for the diatomaceous earth. Such materials as Old Dutch Cleanser, bentonite, rotten stone, wood flour, and Dicalite "4200" were tried.

The results from the calibration tests which were run following the polishing with each of these materials are shown in Table II.

Table II shows the beating time and bursting strength at 250 ml. Canadian Standard freeness resulting from the calibration tests following the polishing with each of the different

**Table II. Smooth Bar Beater Calibration Test Results Using Different Polishing Materials**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* | *Polishing material used.* |
|---|---|---|---|---|
| X-29 | 36 | 105 | 5.0 | Old Dutch Cleanser |
| X-30 | 36 | 103 | 5.0 | Bentonite |
| X-31 | 33 | 104 | 5.0 | Rotten stone |
| X-34 | 32 | 104 | 5.0 | Dicalite "4200" |
| No test | .. | .. | .. | Wood flour |
| X-39 | 35 | 100 | 5.0 | Dicalite "4200" (dammed section) |
| TAPPI $\bar{X}$ | 43 | 114 | 5.5 | TAPPI calibration |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight in kilograms on tackle arm. TAPPI $\bar{X}$ = average of test in TAPPI calibrated beater. Pulp = standard calibration pulp.

materials. The results from the tests following these polishing materials (tests X-29 to X-34) were again much lower in burst than those obtained with the TAPPI calibration (TAPPI $\bar{X}$). The wood flour produced very badly scratched bar and bedplate surfaces so no test was run following this material. The Dicalite "4200" produced the most highly polished surface. Thus, the polishing procedure with the Dicalite "4200" was repeated in the dammed section of the beater which would make possible higher concentrations of the polishing material. Again the test results (Test X-39) showed a low burst as compared to the TAPPI $\bar{X}$ values.

The study to this point had been confined to polished surfaces on the bars and bedplate. I now decided to investigate the effect of varying the surface roughness on strength development.

The procedure for obtaining the desired surfaces on the bars and bedplate was to add 100 grams of the grinding compound to a well-beaten pulp circulation in the beater. The bedplate was then engaged and clamped into position and the grinding was continued until the desired surface was obtained.

First the beater was ground using 80-grit carborundum. Then without rounding the leading edges or removing the burs a calibration test was run in the beater. The beater was then ground using 120-grit carborundum which was followed by a calibration test. Again the procedure was repeated with 200 and finally with 400-grit carborundum. The results from the calibration tests run with these different degrees of surface roughness are shown in Table III.

**Table III. Bursting Strength Development versus Degrees of Surface Roughness**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* | *Surface grit* |
|---|---|---|---|---|
| 240 | 37 | 114 | 5.0 | 80 |
| 241 | 28 | 107 | 5.0 | 120 |
| 242 | 26 | 102 | 5.0 | 220 |
| 243 | 25 | 94 | 5.0 | 400 |
| TAPPI $\bar{X}$ | 43 | 114 | 5.5 | TAPPI |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight in kilograms on tackle arm. Surface grit = carborundum mesh used to obtain the surface on the bar and bedplate. TAPPI X = average of test in a TAPPI calibrated beater. Pulp = standard calibration pulp.

It is interesting to note that Test 240 with the 80-grit surface shows a bursting strength development for the calibration pulp which was comparable to the TAPPI $\bar{X}$ value. Also with each of the other surfaces the burst developed at 250 ml. Canadian Standard freeness became progressively lower with each successively finer bar surface.

At the completion of this series of tests I had a 400-grit surface on the bars and bedplate. Thus, I decided to polish the bars by a different method. The roll was wrapped with 150-grit emery cloth which was secured in place with a wooden wedge. The roll was then set into motion and the bedplate was brought into contact. This produced a semipolished surface on the bedplate bars. Next the roll bars were polished by holding a block of wood wrapped with 150-grit emery cloth against the rotating roll. This produced a semipolished surface on the roll bars. This surface was comparable with the surface obtained when bleached pulp and diatomaceous earth were used for polishing.

With this semipolished surface on the bars and bedplate the effect of varying the roundness of the bars leading edges on bursting strength development was studied. The radius of roundness was varied from $^1/_{64}$ to $^5/_{64}$ in. in $^1/_{64}$-in. increments. These variations were only approximated and a very fine carborundum stone was used to obtain the desired roundness. A calibration test on the standard pulp was run in the beater after each increase in roundness. The results of these tests are tabulated in Table IV.

**Table IV. The Effect on Strength Development of Different Radii of Relief on the Roll Bar's Leading Edges with Smooth Bar**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* | *Edge radius, in.* | *Ratio* |
|---|---|---|---|---|---|
| 256 | 32 | 103 | 5.0 | $^1/_{64}$ | 1 |
| 257 | 35 | 106 | 5.0 | $^2/_{64}$ | 2 |
| 264 | 44 | 109 | 5.0 | $^3/_{64}$ | 3 |
| 263 | 57 | 114 | 5.0 | $^4/_{64}$ | 4 |
| 267 | 53 | 112 | 6.0 | $^5/_{64}$ | 5 |
| TAPPI $\bar{X}$ | 43 | 114 | 5.5 | .. | .. |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight in kilograms on tackle arm. Edge radius = approximate radius in inches on leading edges. TAPPI $\bar{X}$ = average of test in a TAPPI calibrated beater. Pulp = standard calibration pulp.

A study of the results in Table IV indicates that a rather large radius of roundness was needed before the cutting action was sufficiently reduced to give a strength development comparable to TAPPI $\bar{X}$ values.

The bedplate retaining lead at the point of initial contact with the roll bars was observed after each test. The sharp edge at this point was found to wear away very rapidly. Thus, the sharp edge on the lead was considered to be of minor importance.

I had now found two different bar and bedplate conditions which gave strength developments of a comparable value with that obtained for the TAPPI calibrated beater. These conditions were: (1) the 80-grit surface on the bars and bedplate and (2) semipolished surfaces with sufficient leading edge roundness.

Thus, I decided to reinvestigate the 120, 220, and 400-grit surfaces on the bars and bedplate, but this time the leading edges of the roll bars were rounded approximately $^1/_{16}$ in. The test results from this series compared favorably with the results obtained with the semipolished surfaces with a sufficiently rounded leading edge on the roll bars. The results shown in Table V indicate that with sufficient leading edge roundness the bursting strength developed is comparable to the TAPPI $\bar{X}$ values.

**Table V. Bursting Strength Development versus Different Surfaces with Sufficient Leading Edge Relief**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* | *Surface grit* | *Edge radius, in.* |
|---|---|---|---|---|---|
| 240 | 37 | 114 | 5.0 | 80 | No |
| 270 | 42 | 115 | 5.5 | 120 | $^1/_{16}$ |
| 272 | 43 | 113 | 5.5 | 220 | $^1/_{16}$ |
| 273 | 45 | 112 | 5.5 | 400 | $^1/_{16}$ |
| 263 | 57 | 114 | 5.0 | Polished | $^1/_{16}$ |
| TAPPI $\bar{X}$ | 43 | 114 | 5.5 | TAPPI | .. |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight in kilograms on tackle arm. Surface grit = carborundum grit used to develop bar and bedplate surfaces. Edge radius = approximate radius in inches on leading edges. TAPPI $\bar{X}$ = average of test in a TAPPI calibrated beater. Pulp = standard calibration pulp.

I was now confronted with the question, how long would a beater remain in calibration and continue to give uniform reproducible results: (1) with the semipolished surfaces obtained with the 150-grit emery cloth, and the leading edges rounded sufficiently; (2) with the 80-grit surfaces with no leading-edge roundness?

A beater was conditioned having semipolished surfaces. Then a series of calibration tests on the standard pulp were run in the beater to learn how long the beater would remain in calibration. The results from one of these series of tests are presented in Table VI under the section headed No Solvent Wash.

From several series of tests in the semipolished beater I noted that after 10 to 12 tests the bursting strength developed at 250 ml. Canadian Standard freeness began to show lower values. As shown in Table VI, I obtained a definite breaking off point following the 10th test. In addition, the beating time was approaching the upper limit for practical routine testing. During these series of tests considerable pitch accumulation was found on the beater walls. As a result of this observation I reasoned that an accumulation of pitch could also exist on the bar and bedplate surfaces, and could very possibly have an effect on the in-calibration period of the beater.

Thus the beater was reconditioned by the emery cloth polishing, and another series of calibration tests were run in the beater. But during this series the bars and bedplate were thoroughly washed with a solvent such as xylene just prior to each test. The results from this series are shown in Table VI headed Xylene Wash.

From the tabulated results one can see that the xylene wash removed a sufficient quantity of pitch to extend the incalibration period approximately 50%.

A similar investigation was conducted with the beater having an 80-grit surface and no roll-bar leading edge relief. The pattern of in-calibration obtained from this study was very simliar to that found with the semipolished beater.

It now became evident that these slight pitch accumulations were somewhat of an uncontrollable factor. However, the 80-grit surface developed an in-calibration beater without requiring additional conditioning. Therefore, it appeared that the key to the problem was to find a method for maintaining the original 80-grit surface. This seemed to indicate the need for a regular touch-up of the surfaces.

The method proposed for conditioning the beater with the 80-grit carborundum and the periodic touch-ups was as follows:

**Table VI. The Effect of No Solvent Wash versus Solvent Wash on Beating Time and Bursting Strength in Semipolished Bar Beater**

| | *No solvent wash* | | *Xylene wash* | |
|---|---|---|---|---|
| *Test no.* | *Beating time* | *Bursting strength* | *Beating time* | *Bursting strength* |
| 1 | 53 | 113 | 51 | 114 |
| 2 | 56 | 114 | 53 | 112 |
| 3 | 57 | 114 | 53 | 114 |
| 4 | 59 | 115 | 56 | 115 |
| 5 | 59 | 113 | 56 | 113 |
| 6 | 59 | 115 | 58 | 114 |
| 7 | 60 | 115 | 60 | 112 |
| 8 | 61 | 113 | 60 | 112 |
| 9 | 62 | 113 | 61 | 114 |
| 10 | 64 | 112 | 62 | 115 |
| 11 | 64 | 109 | 64 | 114 |
| 12 | 65 | 108 | 63 | 112 |
| 13 | 64 | 109 | 63 | 114 |
| 14 | 65 | 108 | 64 | 113 |
| 15 | .. | .. | 65 | 113 |
| 16 | .. | .. | 64 | 110 |
| 17 | .. | .. | 65 | 109 |
| 18 | .. | .. | 67 | 108 |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Pulp = standard calibration pulp.

***Conditioning***

Furnish the beater with a charge of well-beaten bleached pulp (300 ml. Canadian Standard freeness), add 100 grams of 80-grit carborundum to the pulp circulating in the beater with the bedplate disengaged. Engage the bedplate after 5 min. of circulation, and clamp the bedplate lever arm into position during the grinding. Continue the grinding until the proper fit between the bedplate and bars is obtained as shown by an impression. When long grinding periods are required an additional 10 grams of 80-grit carborundum are added at 5-min. intervals. At the completion of the grinding all of the abrasive material is thoroughly washed from the beater.

The beater is now charged with a furnish of bleached pulp which is allowed to beat for 1 hr. This is repeated with two additional charges of bleached pulp. Following the slowing down the beater is charged with a standard bleached pulp and checked for beating time and strength development. The proper beating time to 250 ml. Canadian Standard freeness was 40 to 50 min. for the standard pulp. Additional adjustments to the beating time can be made by varying the weight on the tackle arm.

After completing the conditioning of the beater it is maintained by regular touch-ups after every eighth beater test, or when the beating time for the standard pulp goes beyond 50 min. to 250 ml. Canadian Standard freeness.

***Touch-Up***

The touch-up was performed when needed by adding 7 to 10 cc. of 80-grit carborundum to the pulp remaining in the beater at the completion of a beater test. The pulp and carborundum were allowed to circulate for 5 min. to assure good mixing before engaging the bedplate. The bedplate was then engaged in 1-sec. intervals. The number of seconds of engagement are determined from the desired decrease in beating time. Each second of touch-up will reduce the beating time to 250 ml. Canadian Standard freeness by 3 to 5 min.

The outlined procedure for conditioning and the periodic touch-up of the beaters has been in use more than 18 months with excellent results.

The remaining variable to be studied was the effect of varying the contact pressure by varying the bedplate lever arm weight. This study was made on the semipolished and the 80-grit surfaced beater. The net weight on the lever arm was varied between 4.5 and 6.5 kg. The results from this study are shown in Table VII.

**Table VII. The Effect of Varying Contact Pressure on Strength Development**

| *Test no.* | *Beating time* | *Bursting strength* | *Weight* | *Bar surface* |
|---|---|---|---|---|
| 1-A | 53 | 113 | 6.0 | Semipolished |
| 1-B | 57 | 115 | 5.5 | Semipolished |
| 1-C | 55 | 112 | 5.0 | Semipolished |
| 2-A | 43 | 114 | 6.0 | Semipolished |
| 2-B | 44 | 112 | 5.0 | Semipolished |
| 2-C | 50 | 113 | 4.5 | Semipolished |
| 3-A | 35 | 116 | 6.5 | 80-grit |
| 3-B | 40 | 114 | 5.5 | 80-grit |
| 3-C | 48 | 114 | 4.5 | 80-grit |
| 4-A | 42 | 114 | 6.5 | 80-grit |
| 4-B | 46 | 112 | 5.5 | 80-grit |
| 4-C | 50 | 113 | 4.5 | 80-grit |

Beating time = beating time, min., to 250 ml. Canadian Standard freeness. Bursting strength = points at 250 ml. Canadian Standard freeness. Weight = weight in kilograms on tackle arm. Bar surface = semipolished or 80-grit surface on bars and bedplate. Pulp = standard calibration pulp.

The results from the contact pressure study indicate that no significant difference in strength development was noted within the range of weight variations studied.

The data accumulated from the numerous calibration tests run during this investigation were tabulated to determine the

reproducibility of each method of calibration. The results of this tabulation are summarized in Table VIII.

**Table VIII. A Comparison of the Test Results of the Three Methods of Beater Calibration Studied**

| | *Original* | *Semipolished* | *80-grit* |
|---|---|---|---|
| Number tests | 57 | 45 | 246 |
| 250 cc. Canadian Standard freeness bursting strength | | | |
| High | 124[a] | 121[a] | 120[a] |
| Low | 106[a] | 110[a] | 110[a] |
| Average $\overline{X}$ | 113 | 114 | 114 |
| Range | 18 | 11 | 10 |
| Range above mean + | +11 | +7 | +6 |
| Range below mean − | −7 | −4 | −4 |

[a] The high and low values are from calibration tests including times that the beater was out of calibration and was not used for commercial testing.

From the data shown in Table VIII it is evident that the range of 18 points of burst, obtained when calibrating the beater using the original method of calibration, made it very difficult to bring the beater to the desired condition. With such a wide range of calibration results possible it was often necessary to repeat the operation of calibrating the beater several times before the strength development desired for control testing was obtained. This meant that frequently the beaters had to be removed from routine control testing and had to be reconditioned to obtain the proper calibration before the beater could be placed back into the control testing program.

When using the semipolished bar method the range of test values was much narrower (11 points of burst). This method also required a great deal of reconditioning and frequent removal of the beater from control testing.

With the 80-grit touch-up method the range was found to be 10 points of burst. This was only a slight improvement in reproducibility over the semipolished bar method, but an additional unexpected benefit was the reduction in beater maintenance.

The 80-grit touch-up method has been in use over 18 months. It has continued to give the same degree of reproducibility along with the reduction in beater maintenance. Thus, test results have become more significant, and more time has been available to study other problems.

The data and results from this study have only been presented in view of the effect on the bursting strength development, but much the same was found to be true for the other properties studied such as fold, tear, and density.

The total wear on the bars and bedplate when using the touch-up method has been surprisingly small. After more than 18 months of use of the 80-grit touch-up method no serious problems have presented themselves.

## CONCLUSION

I would like to sum up the findings of this investigation as follows:

1. The bedplate should be properly placed and have a correctly shaped lead grout to give proper and uniform calibration test results.
2. The roughness of the bar and bedplate surfaces does not affect the strength development providing the bar's leading edges are sufficiently rounded.
3. Varying the weight on the bedplate lever arm within reasonable limits has no effect on strength development.
4. The total mechanical wear on the bedplate and bars using the 80-grit touch-up method is negligible.
5. The 80-grit surface on the bars and bedplate is of such a roughness that the bars' leading edges are sufficiently rounded to give the proper strength development.
6. The slight pitch accumulation on the beating surfaces was found to be an uncontrollable factor with other method of conditioning and calibrating the beaters.
7. The 80-grit touch-up method has eliminated the problem of pitch build-up on the beating surfaces.
8. The degree of reproducibility when using the 80-grit touch-up method has been an improvement over the old method, and has continued to give uniform and reliable results.
9. In the majority of cases a failure to achieve normal calibration has been traced to improper technique or to the test equipment following the beater.

Now when I am asked, "Are the beaters in calibration?," a review of the routine calibration data, in almost every instance, indicates "yes," the beaters are in calibration.

# Methods and Results of a Kraft Mill Sodium Balance

MAX L. CUSTIS

THE total sodium sulphate loss from a kraft mill is continuously checked by the amount of salt cake it is necessary to add to the system to maintain a desired operating inventory. The measurements of the individual losses are usually incomplete and are a source of much speculation. It was decided that a complete sodium balance should be made on the Camas kraft mill in order to pinpoint with some accuracy the major losses and the proportionate amounts contributed by individual sources. It was felt that savings might be possible through changes in operating details in some places where high losses indicated the need.

## PLAN OF TESTING

The sodium content at the three major points of loss: the furnace stack gases, the pulp leaving the mill, and the main sewer. were determined first. After a reasonable agreement was obtained between these three measured components, and the total loss as determined by salt cake make-up and inventory changes, the losses in the individual components of the main sewer were estimated to discover if any of them were excessive.

### *Summary of Sodium Balance*

Sodium balance data on the three major sources are shown

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*Fig. 1. A photograph of equipment used on the furnace stack*

**Table I. Summary of Sodium Balance**

| Dates | Inventory change, $Na_2SO_4$ lb./day | $Na_2SO_4$ added, lb./day (lb./ton) | Net loss $Na_2SO_4$, lb./day (lb./ton) | $Na_2SO_4$ to sewer, lb./day (lb./ton) | $Na_2SO_4$ in pulp, lb./day (lb./ton) | $Na_2SO_4$ stack loss, lb./day (lb./ton) | Total measured losses, lb./day (lb./ton) | Error, % |
|---|---|---|---|---|---|---|---|---|
| 11/6–12/11/53 | +1160 | 41,900 (98) | 40,740 (95.3) | 14,390 (32.8) | 16,510 (38.6) | 9910 (23.1) | 40,810 (95.5) | +0.2 |
| 12/31–1/13/54 | +4270 | 48,300 (113.5) | 44,030 (104.0) | 12,970 (30.5) | 20,760 (48.8) | 9700 (22.8) | 43,430 (102.0) | −1.3 |

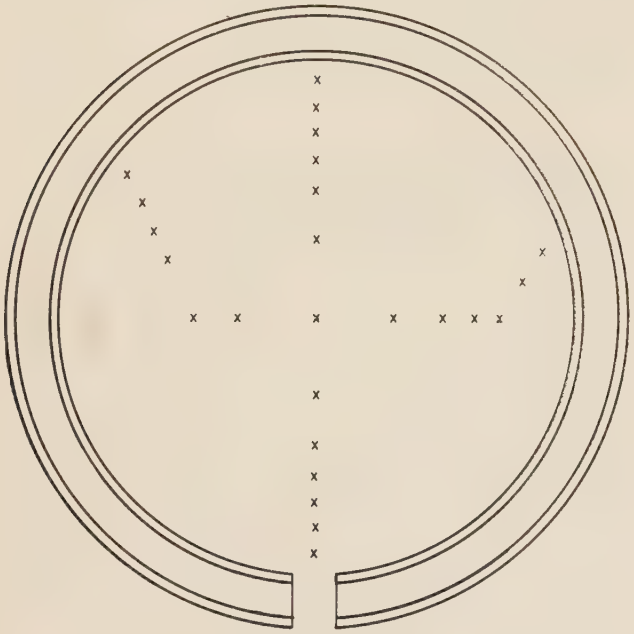

***Fig. 2. Cross section of furnace stack showing sample points***

in Table I. The data on sewer and pulp losses are averages of daily determinations. Stack losses were determined bimonthly. The data between the two periods shown were not reported because they were discontinuous and because inventory was changing rapidly, making accurate determination of losses impossible. During the periods covered, the data on losses at the three points tested accounted for over 98% of the total mill losses. When shorter time periods were covered, the accuracy was decreased to about 85 to 90%.

Several sources of loss, such as slaker dregs, lime kiln stack dust, and one small ditch sewer of discontinuous flow, were not included because they were known to be quite small.

## FURNACE STACK LOSSES

Salt cake losses from the furnace stack were obtained from gas volume and gas dust concentration measurements, using methods described in Western Precipitation Bulletin WP-50.

Since accurate measurements of velocity and dust load can only be made in long straight sections of gas ducts, a platform was erected 100 ft. up on the 140-ft. furnace stack, as shown in Fig. 1. At this point a small rectangular opening was cut through the brickwork to permit insertion of the testing equipment. The Pitot and dust tubes were mounted together and fitted with long extensions for reaching across the 12 ft. inside diameter of the stack. The condensation of the stack gases in the sample thimble was thus avoided since the entire unit was maintained at the stack temperature (about 300°F.).

The test procedure consisted of taking stack gas samples from the platform for a period of about 4 hr. The stack gas was drawn through a paper extraction thimble at a rate determined by the gas velocity; the latter being calculated from draft gage readings of the Pitot tube. To assure a representative test, 10-cu. ft. gas samples were taken at 25 points located at equal area mid-points, as shown in Fig. 2. Space limitations of the platform and opening prevented the use of two right-angle traverses which would have been a little more desirable. The dust filtered from the stack gas was taken as a measure of the loss. Test data obtained are shown in Table II.

Previous data on Cottrell operation indicate it should be operating at approximately one-third less total loss than is presently sustained. Investigation has shown that there is a tendency for all dust to go through the last two units, leaving the first two with subnormal loads. Close attention to damper control has helped to alleviate this condition.

In order to keep normal losses as low as possible and to detect failure in the Cottrell operation as soon as possible, an improvised photocell arrangement was installed in the ductwork between the Cottrell precipitator and the stack. A recording meter was installed to give a continuous record of the amplified photocell current. This meter was roughly calibrated by stack dust loss measurement as described above, sections of the Cottrell unit being shut down for a short time in order to vary the dust loss. This system proved helpful in detecting trouble in the Cottrell operation, and has recently been replaced by a commercial dust detector unit which is operating satisfactorily.

## PULP SODA LOSS MEASUREMENTS

Pulp soda losses are measured at Camas by determining the sodium concentration of the pulp filtrate with a conductivity cell which has been calibrated with black liquor. The sodium concentration in the filtrate is assumed the same as that in the filtrate remaining in the filtered pulp. It is also assumed that the amount of sodium attached to the fiber is not significant. The validity of these assumptions will be shown later. Calculations for soda loss per ton of pulp are made from the filtrate sodium concentration and the consistency of the pulp sample, as shown below:

$$\frac{\text{Lb. } Na_2SO_4}{\text{Ton m.f. pulp}} = \frac{\text{g. } Na_2SO_4}{1000 \text{ g. filtrate}} \times \frac{\text{g. slurry} - \text{g. pulp}}{\text{g. pulp}} \times \frac{2000 \text{ lb.}}{\text{ton}}$$

This is a routine test run on grab samples taken at the wash-

**Table II. Furnace Stack Loss Data**

| Test date | Gas volume, cu. ft./min. | Dust loss, lb./hr. | Dust conc., grains/cu. ft. wet gas | Equiv. loss $Na_2SO_4$, lb./day | Equiv. loss, $Na_2SO_4$/lb. air-dry ton pulp |
|---|---|---|---|---|---|
| 10/16/53 | 152,000 | 317 | 0.243 | 8,500 | 19.8 |
| 12/2/53 | 161,000 | 418 | 0.303 | 11,250 | 26.2 |
| 2/5/54 | 137,000 | 360 | 0.311 | 9,700 | 22.6 |

*Note:* Equivalent losses are based on total sodium determination of 1.12 grams $Na_2SO_4$ per gram of dust.

Table III. Component Sodium Losses of the Main Sewer

| *Sample point* | *Flow rate, gal./day* | *Grams $Na_2SO_3$/liter* | *$Na_2SO_4$ loss, lb./day* | *$Na_2SO_4$ loss, lb./air-dry ton pulp* |
|---|---|---|---|---|
| Washer sewer | 214,000 | 1.25 | 2,230 | 5 |
| Washer vacuum pumps | ... | 0.0018 | ... | ... |
| Swensen condensate sewer | 798,000 | By diff. | 1,585 | 4 |
| Caustic room sewer | 1,170,000 | 2.815 | 7,260 | 17 |
| —before Zaremba evaporators | 166,900 | 1.472 | 540 | 1 |
| —Zaremba evaporators | 1,004,000 | By diff. | 6,730 | 16 |
| —Zaremba evaporators reduced vacuum | 1,004,000 | 1.215 | 2,690 | 6 |
| Main sewer | 3,100,000 | 1.860 | 12,700 | 29.5 |

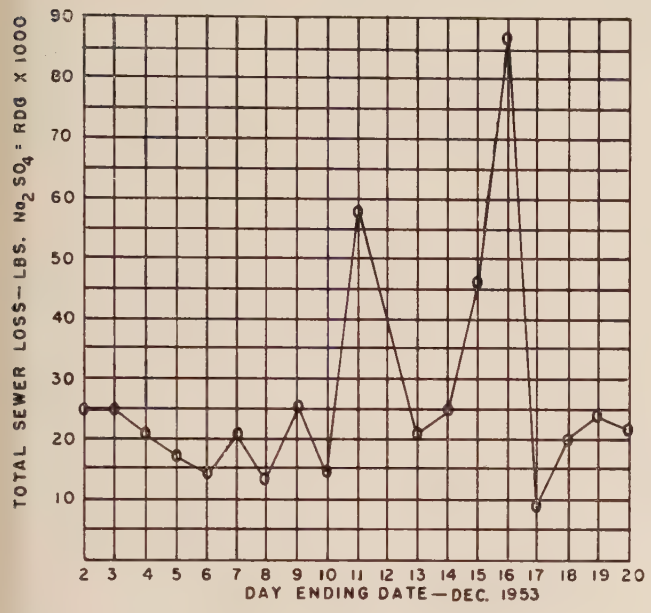


*Fig. 3. Chart of total sewer losses for December, 1953*

ers once a shift. During steady operation it provides satisfactory results, but during periods of fluctuation it is easily seen that in a sample taken once a shift may not be truly representative of every day.

## SEWER LOSS DETERMINATIONS

The flow rate through the kraft mill main sewer was obtained by building a weir in the sump receiving the sewer and installing a recording level meter behind it. The data obtained had an estimated error of 10% due to the limitations of the level recorder. A power-driven sampler was used to obtain a daily composite sample on which sodium determinations were run. Results for part of the month of December are shown in Fig. 3. Peak losses on the eleventh and sixteenth occurred during short runs of cottonwood, which is particularly difficult to wash clean.

The losses of the individual sewers entering the main sewer were roughly estimated by spot checks. Some valuable information was obtained, but accurate measurement will require composite samples and a continuous record of flow since losses are known to vary considerably through the day.

Figure 4 shows a sketch of the sump receiving the components which make up the main sewer, except for hot water overflow. Determined losses are listed by each sewer. Flow measurements in components sewers were made with weirs where possible. Other flow rates were estimated by measuring surface velocities and by use of Manning's formulas for partially filled channels. Sodium concentration in all sewer samples was determined by a Beckman model DU flame spectrophotometer.

The data obtained (see Table III) indicate the Zaremba evaporators which feed into the caustic room sewer are the major source of continuous loss. Reduction of the vacuum caused this loss to drop to about one third of the original value. The results obtained on the Swensen condensate sewer were confirmed by observation of its color. The loss here is in the form of black liquor, and periodic checks indicated it was continuously light in color. The losses in the washer sewer are known to vary considerably due to occasional operating difficulties in washer and knotter operation. Losses shown here are not very revealing as to either maximum or average losses at this point. Losses in this sample are shown to be largely from the washers and knotters by the low concentration obtained on the vacuum pump effluent, the other component of this sewer.

## ANALYTICAL PROCEDURES

It was decided at the beginning of this investigation that a check should be made of the analytical procedures used in determining component losses, using the TAPPI Standard T 623 m-44 (uranyl zinc acetate method) as the referee method.

The sodium concentration as determined by conductivity measurements was checked by tests made on a sample of weak black liquor. Results of these tests are shown in Table IV. These show the conductivity determination checks with TAPPI Standard T 625 m-44 by which the conductivity cell was calibrated, but is about 7.5% above the referee value for total sodium. This error was subsequently corrected in the calculation of daily pulp soda losses.

Table IV. Total Sodium Determinations on Weak Black Liquor

| *Test method* | *Grams $Na_2O$/liter* |
|---|---|
| Conductivity | 42.78 |
| TAPPI Std. T 625 m-44 | 42.54 (calibration method for conductivity cell) |
| TAPPI Std. T 623 m-44 | 39.23 |

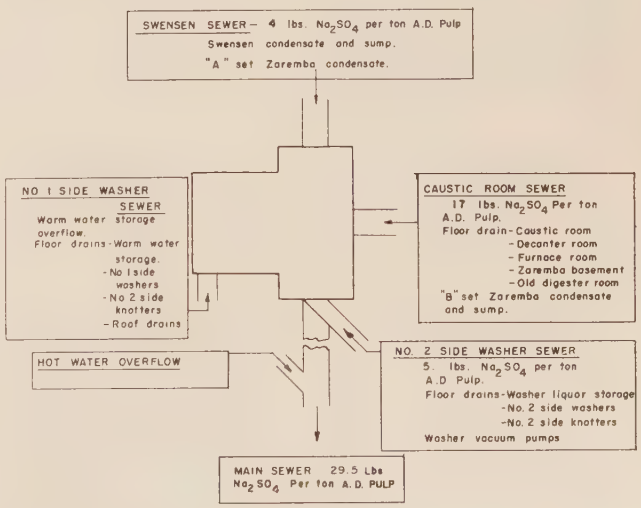
SWENSEN SEWER— 4 lbs. $Na_2SO_4$ per ton A.D. Pulp
Swensen condensate and sump.
"A" set Zaremba condensate.
NO. 1 SIDE WASHER SEWER
Warm water storage overflow.
Floor drains-Warm water storage.
-No. 1 side washers
-No. 2 side knotters
-Roof drains
CAUSTIC ROOM SEWER
17 lbs. $Na_2SO_4$ Per ton A.D. Pulp.
Floor drain-Caustic room
-Decanter room
-Furnace room
-Zaremba basement
-Old digester room
"B" set Zaremba condensate and sump.
HOT WATER OVERFLOW
NO. 2 SIDE WASHER SEWER
5. lbs. $Na_2SO_4$ per ton A.D. Pulp.
Floor drains-Washer liquor storage
-No. 2 side washers
-No. 2 side knotters
Washer vacuum pumps
MAIN SEWER 29.5 Lbs $Na_2SO_4$ Per ton A.D. PULP

*Fig. 4. Sump receiving individual sewers to form main sewer*

The possibility that a significant amount of sodium might be attached to the fiber was also investigated. A pulp sample was washed repeatedly until the sodium content was reduced to a very low value. It was reasoned that any soda not measurable by conductivity would still be attached to the fiber and nearly identical samples could be obtained for the two methods of testing by splitting the sample prepared in this manner. Results of tests for sodium content of this pulp by the referee method and the conductivity method (shown in Table V) indicate a reasonable agreement between the two methods. Therefore, the presence of any sodium on the fiber of samples was ignored.

**Table V. Accuracy of Soda Losses by Conductivity**

| *Test method* | *Pounds $Na_2SO_4$/ton air-dry pulp* Hemlock | Douglas-fir |
|---|---|---|
| TAPPI Std. T 625 m-44, T 623 m-44 | 5.74 | 3.98 |
| Conductivity | 5.75 | 5.38 |

The use of the conductivity tests on sewer samples was tried with fair success but it was found that the conductivity cell soon became covered with grease from the sewer and the cell calibration was destroyed.

A Beckman flame spectrophotometer was next tried on the sewer sample. This was tried on black liquor and on a sewer sample in comparison with the referee method. Results obtained in these tests are shown in Table VI. In these tests

**Table VI. Sodium Determinations by flame spectrophotometer**

| *Test method* ⟶ *Sample tested* | *Flame spectrophotometer Grams $Na_2O$/liter* | *TAPPI Std. T 623 m-44 Grams $Na_2O$/liter* |
|---|---|---|
| Weak black liquor | 37.8 | 39.2 |
| Sewer sample, daily composite | 0.30 | 0.317 |

the flame spectrophotometer was about 3% lower than the referee method. No correction was made for this difference.

## CONCLUSIONS

The purpose in gathering these data was to identify the sodium losses from the three major sources of loss with a reasonable degree of accuracy and further to check the component losses of the main sewer to identify any excessive losses. It is believed that these objectives have been obtained. Expressed as pounds sodium sulphate per ton of air-dry pulp, furnace stack losses were 20 to 25 lb.; pulp soda losses were 40 to 45 lb.; and sewer losses were about 30 lb. The major sources of loss in the main sewer were the Zaremba evaporators and the washer and knotter operations.

# Possible Improvements in the Routine Analysis of Ordnance Pulp

J. F. FARRINGTON

The alpha-cellulose, beta-cellulose, and gamma-cellulose fractions, as designated by Cross and Bevan (*1*), are important factors in determining ordnance pulp quality. These fractions may be analyzed volumetrically or gravimetrically, but for this problem of testing ordnance pulp the gravimetric method is required for alpha-cellulose and beta-cellulose with gamma-cellulose being calculated by difference.

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The methods are described in detail in JAN-C-216 and in PA-PD-153. The sections pertaining to the analysis of alpha, beta, and gamma-cellulose may be seen in Appendix A. Each type of cellulose must be present in prescribed amounts for ordnance pulp to meet chemical requirements. The gravimetric method is empirical and to obtain duplicate and accurate results the conditions of the test must be followed exactly.

## DISCUSSION OF PROBLEM

In the use of the required analytical methods for routine testing of the cellulose fractions, the agreement between answers for a common sample varied sufficiently to necessitate repeat determinations rather frequently. This situation increased the testing time required and restricted the degree to which these values could be used for control of pulp quality. Many of the variables which affected the alpha-cellulose and beta-cellulose results were related to the filtering of the respective fractions.

### *Alpha-Cellulose*

To filter the alpha-cellulose, a finely perforated crucible was recommended which allowed passage of some of the fibers. This necessitated refiltering of the filtrate for collection of these fibers on the alpha-cellulose pad formed in the initial separation. Duplicate results were obtainable by this procedure, but at the expense of testing time. By using coarse sintered glass funnels in lieu of the crucibles, reproducible values were frequently achieved with a decrease filtering time. However, the accuracy of the alpha-cellulose separation was questionable because of the necessity of transferring the alpha pad from the funnel back to the original weighing bottle for drying and weighing. After transferring the alpha pad, the funnel was treated with cleaning solution to remove any remaining alpha fibers. This was an additional step in the testing procedure which could be eliminated. An improved filtering medium was necessary to obtain reproducible and accurate results with increased testing efficiency.

### *Beta-Cellulose*

A similar filtering situation existed in the beta-cellulose determination. The testing procedure stated that a 9-cm. no. 41 Whatman filter paper or a similar type be used for collecting the beta precipitate. Results show that these filter papers are questionable for the filtration of beta-cellulose since there is an indication that gamma-cellulose is absorbed by the filter medium (*2*). If the filter papers and beta-cellulose precipitate are thoroughly washed, good correspondence between values for duplicate samples is found. However, the routine control tests required more time because the beta-cellulose precipitate frequently clogged the papers to the extent that the filtering rate was very slow during the washing.

## INVESTIGATION

To eliminate the difficulties encountered in the alpha and beta-cellulose determinations, a study for possible improvement in the routine analysis of ordnance pulp was initiated. This study included many of the physical and chemical variables which might influence the results. Particular emphasis was placed on various filtering media for a more effective separation of the two cellulose fractions.

The question of sample preparation was not included in this paper because of the range of the problems related to that particular phase. Sample drying conditions were checked because alpha and beta-cellulose are calculated as per cent of the dried pulp sample weight and a reliable value was necessary. For removing moisture from pulp samples, the required drying conditions of 2 hr. and 100 to 105°C. were sufficient. Samples dried under these conditions gave practically constant weights, but they showed a slight decrease in weight due to

further moisture loss over extended drying periods. However, the increased loss was found to be negligible in calculating the fraction percentages.

### *Alpha-Cellulose*

The empirical nature of the routine gravimetric determination of ordnance pulp required conformity to the test conditions. For the analysis of the alpha-cellulose fraction, the weighed dried pulp sample was transferred from the weighing bottle and treated with 25 ml. of carbonate-free 17.5% sodium hydroxide. After 45 min. in a water bath (24.5°), 40 ml. of distilled water were added to the beaker and the contents mixed thoroughly. The mixture was filtered through a tared coarse sintered glass crucible. The alpha-cellulose pad was washed with 20 ml. of 10% acetic acid to neutralize the remaining caustic and this was followed with 250 ml. of distilled water. This alpha filtrate was used for the beta-cellulose determination. The alpha-cellulose pad and sintered glass crucible were placed in the original sample weighing bottle and the bottle put in the oven for 8 hr. drying time at 100 to 105°C.

The use of this procedure in routine testing leads to satisfactory agreement between duplicate tests. While the effect of departing from the procedure was not studied in detail, a brief investigation was made of the 10% acetic acid rinse of the alpha pad. In a group of 15 alpha tests, seven pads were rinsed with 15 ml. of 10% acid and the remaining eight were washed with 25 ml. The 15 samples were taken from a common source and were dried and tested in accordance with the above procedure except for the rinse.

Results of the tests are shown in Table I.

**Table I. 10% Acetic Acid Rinse**

| | *Fifteen ml. alpha-cellulose* | *Twenty-five ml. alpha-cellulose* |
|---|---|---|
| | 92.87 | 92.76 |
| | 92.78 | 92.84 |
| | 93.00 | 92.89 |
| | 93.00 | 92.86 |
| | 92.79 | 92.80 |
| | 92.82 | 92.80 |
| | 92.86 | 92.82 |
| | | 92.81 |
| Av., % | 92.87 | 92.82 |

There were no significant differences caused by washing the alpha-cellulose pad with various quantities of 10% acetic acid. The agreement between alpha-cellulose results was very close for the 15 tests using the coarse sintered glass crucibles, a range of 0.24% being noted for the above results. The coarse sintered glass crucibles retained the fibers on the initial separation and thereby eliminated refiltration of the solution.

### *Beta-Cellulose*

Beta-cellulose is designated as the portion of cellulose dissolved in 17.5% sodium hydroxide at 24.5 ± 0.3°C. and reprecipitated in acid solution. The amount of beta-cellulose precipitated upon acidification of the alpha filtrate is a quantitative measure of the beta present in a pulp sample. To study the effects of beta-precipitation, eight 300-ml. samples were pipetted from a uniform solution of alpha filtrate and treated with varying excess quantities of acetic acid.

The acid was added to the alpha filtrate in slightly different manners to determine if any significant changes would result. In Table II the odd-numbered samples were stirred, neutralized, and treated with corresponding amounts of excess acid. To the even-numbered samples, the excess quantities of acid and the volume needed for neutralization of the caustic were added as a unit with agitation.

The acidified filtrate was heated to 95 ± 5°C. and digested at this temperature for 1 hr. on a hot plate. The beta precipitate was allowed to settle and filtered while still hot (80 to 90°C.) through a Gooch with an asbestos mat (*2*). Each Gooch was washed, dried at 100 to 105°C. for 4 hr., cooled, and weighed. The results of precipitating the beta-cellulose fraction are shown in Table II.

**Table II**

| *Excess HAC, ml.* | *Sample no.* | *Grams of beta ppt.* | *Average, weight in grams* |
|---|---|---|---|
| 5 | 1 | 0.0154 | 0.0160 |
| | 2 | 0.0165 | |
| 10 | 3 | 0.0163 | 0.0166 |
| | 4 | 0.0169 | |
| 20 | 5 | 0.0173 | 0.0164 |
| | 6 | 0.0154 | |
| 30 | 7 | 0.0162 | 0.0161 |
| | 8 | 0.0160 | |

The experimental results indicate the volume of acetic acid used for acidifying the filtrate need only be in slight excess of the amount needed for neutralization of the caustic for complete beta-cellulose precipitation. No significant differences between results could be traced to the methods of acidification when an excess of acetic acid was present in the alpha filtrate. To check on additional beta precipitation, the gamma solutions were heated to 95 ± 5°C. and redigested for 1 hr. After standing overnight, there was no additional beta precipitate visible upon inspection of the solutions. A filtering medium which could retain the small quantities of beta-cellulose precipitate and be easily washed free of possibly absorbed gamma-cellulose was necessary for accurate and reproducible values. Gooch crucibles with asbestos mats retained the beta precipitate and did not absorb gamma-cellulose as pointed out by Lea (*2*). The objection to using this filter in routine control tests was the frequent loss of asbestos during the filtering process because of improper preparation of the mat. A number of media were tested and a few gave duplicate and accurate results. One which proved reliable and easy to prepare was a no. 4 Gooch crucible with a 2.1-cm no. 42 Whatman filter paper and fiberglas mat. The paper retained the beta fraction and the fiberglas prevented the paper from becoming clogged by the precipitate.

To illustrate the reproducibility of beta-cellulose results by using this filtering medium, 15 tests were made by acidifying the filtrates from 15 alpha-cellulose determinations. Since the method of acidification was shown not to produce any significant difference in precipitating beta-cellulose, the recommended 20 ml. of acetic acid were added as a unit with agitation. The acidified filtrate was heated to 95 ± 5°C. and digested at this temperature for 1 hr. on a hot plate. After allowing the precipitate to settle, it was filtered (80 to 90°C.). The filter was washed with approximately 100 ml. of hot water (80 to 90°C.). After drying for 4 hr. the filters were weighed and the percentage of beta-cellulose in each sample was calculated. The results are shown in Table III.

**Table III**

| | | |
|---|---|---|
| 1.65% | | |
| 1.73% | | |
| 1.68% | | |
| 1.62% | | |
| 1.64% | Av. | 1.67% |
| 1.63% | | |
| 1.70% | High | 1.75% |
| 1.70% | | |
| 1.67% | Low | 1.62% |
| 1.63% | | |
| 1.68% | Range | 0.13% |
| 1.70% | | |
| 1.64% | | |
| 1.75% | | |
| 1.65% | | |

After filtering the beta-cellulose fraction, an additional weight increase was noted which could be removed by washing. This increased weight was due to extraneous materials or possibly gamma-cellulose absorbed by the filtering medium. To determine the amount of water necessary for removing this

**Table IV**

| *Volume of wash water used (80–90°C.), ml.* | *Change in weight of filters, gram* |
|---|---|
| 0 | +0.0716 |
| 50 | +0.0018 |
| 100 | +0.0002 |
| 150 | +0.0003 |
| 200 | +0.0001 |
| 250 | +0.0001 |
| 300 | +0.0001 |
| 350 | +0.0001 |
| 400 | +0.0001 |

material, a number of weighed filters were washed with different volumes of water after 420 ml. of gamma solution had been transferred to each filter. The filtering mat for each Gooch crucible was comprised of a 2.1-cm. no. 42 Whatman filter paper and approximately 1 gram of fiberglas. It was found that the crucible weights varied according to the volumes of wash water used. The results showed that approximately 100 ml. of water (80 to 90°C.) was sufficient for washing the Gooch free of the extraneous materials.

Results in Table IV show the weight change of each filter after being treated with gamma solution and the specified volumes of water.

## DISCUSSION OF RESULTS

Reproducible and accurate results were obtained by using the modified filters which increased testing efficiency. In the alpha separations, the coarse sintered glass crucibles gave very good results with a minimum of handling of the alpha pad. The retention of alpha fibers eliminated the necessity of refiltering the solution and eliminated possible error through fiber loss.

A more uniform and rapid filtering time was found by using no. 4 Gooch crucibles with no. 42 filter paper and fiberglas

**Table V**

| | *Original testing procedure (JAN-C-216)* | | *Modified testing procedure* | |
|---|---|---|---|---|
| | *% Alpha* | *% Beta* | *% Alpha* | *% Beta* |
| | 92.21 | 3.10 | 92.45 | 1.65 |
| | 92.40 | 2.87 | 92.43 | 1.73 |
| | 92.53 | 3.29 | 92.40 | 1.68 |
| | 92.53 | 2.58 | 92.48 | 1.62 |
| | 92.60 | 3.10 | 92.52 | 1.64 |
| | 92.46 | 2.85 | 92.37 | 1.63 |
| | 92.75 | 3.16 | 92.29 | 1.70 |
| | 92.59 | 3.27 | 92.52 | 1.70 |
| | 92.55 | 2.48 | 92.45 | 1.67 |
| | 92.75 | 3.11 | 92.43 | 1.63 |
| | 92.62 | 2.43 | 92.53 | 1.68 |
| | 92.44 | 2.32 | 92.37 | 1.70 |
| | 92.78 | 2.32 | 92.44 | 1.64 |
| | 92.53 | 3.10 | 92.43 | 1.75 |
| | 92.45 | 2.95 | 92.42 | 1.65 |
| Av. | 92.54 | 2.86 | 92.44 | 1.67 |
| High | 92.78 | 3.29 | 92.53 | 1.75 |
| Low | 92.21 | 2.32 | 92.29 | 1.62 |
| Range | 0.57 | 0.97 | 0.24 | 0.13 |

mats. Seven or eight minutes were needed to filter 420 ml. of solution. For a corresponding volume, the filtering period for 9-cm no. 41 Whatman filter papers varied according to the degree the beta precipitate clogged the paper. On an average, approximately 12 to 15 min. were required for filtering and washing, with frequent periods of 20 min. being encountered.

The testing procedure (JAN-C-216) was altered to the extent of changing the original filters for those described above. A description of the modified testing procedure may be seen in Appendix B.

To show the agreement between results obtained by using the original and modified procedures, 30 routine control tests were made on ordnance pulp samples. Fifteen samples were analyzed according to each procedure. All samples were taken from the same source and the test results are shown in Table V.

Per cent gamma-cellulose was calculated by subtracting the sum of per cent alpha and per cent beta-cellulose from 100%.

To check the beta-cellulose values determined by using the modified testing procedure, three groups of analyses were made on pulp taken from the same source as the preceding test samples. Gravimetric and volumetric methods were used for analyzing the three groups. Analysis of the first group followed the modified gravimetric procedure. The second group was tested by the same method as the first group, except the beta-cellulose was precipitated by sulphuric acid instead of acetic acid. The use of sulphuric acid eliminated the error caused by acetic acid in determining the gamma-cellulose fraction volumetrically. A volumetric method (*2*) produced the beta and gamma-cellulose values in the third group. Results of the various methods are listed in Table VI.

**Table VI**

| *Fraction* ⟶ | *Alpha* | *Beta* | *Gamma* |
|---|---|---|---|
| Modified Gravimetric Method. Remarks: Using acetic acid to precipitate beta-cellulose | | | |
| | *Grav.* | *Grav.* | *Difference* |
| | 92.24% | 1.88% | 5.88% |
| | 92.32% | 1 67% | 6.01% |
| | 92.22% | 1.71% | 6.07% |
| | 92.36% | 1.69% | 5.95% |
| | 92.36% | 1.69% | 5.95% |
| Av. | 92.30% | 1.73% | 5.97% |
| Modified Gravimetric Method. Remarks: Using 50% sulphuric acid to precipitate beta-cellulose in order to determine gamma-cellulose volumetrically | | | |
| | *Grav.* | *Grav.* | *Vol* |
| | 92.40% | 1.63% | 5.15% |
| | 92.41% | 1.51% | 5.43% |
| | 92.43% | 1.60% | 5.49% |
| | 92.42% | 1.57% | 5.75% |
| | 92.68% | 1.69% | 5.63% |
| Av. | 92.47% | 1.60% | 5.49% |
| Volumetric Method | | | |
| | *Grav.* | *Vol.* | *Vol.* |
| | 92.39% | 1.70% | 5.34% |
| | 92.39% | 1.72% | 5.38% |
| | 92.44% | 1.45% | 5.57% |
| Av. | 92.41% | 1.62% | 5.43% |

## SUMMARY AND CONCLUSION

A review of the routine control analyses of ordnance pulp, as performed in accordance with the prescribed testing procedure (JAN-C-216), indicated desirability of determining whether any modifications could be found for use in the routine control of pulp quality which might enhance its value. The study which was made showed that the filtering operations were primarily responsible for the difficulties encountered. By modifying the testing procedure to provide improved filtering equipment and techniques, reproducible and accurate results were obtained with an increase in testing efficiency.

## LITERATURE CITED


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## APPENDIX A

Chemical requirements: alpha-cellulose 90.00%, beta-cellulose 4.00%, and gamma-cellulose 6.00%.

### *Alpha-Cellulose*

Prepare a portion of the sample by shredding or fluffing. Transfer approximately 1 gram of the material to a tared weighing dish approximately 3 in. in diameter and equipped with a tight-fitting cover, and heat at 100 to 105°C., for 2 hr. with cover removed. Replace the cover, cool in a sulphuric acid desiccator, and weigh. Carefully transfer the weighed sample to a 250-ml. beaker, add 25 ml. of a carbonate-free 17.5% sodium hydroxide solution at 24.5 ± 0.3°C. Place the beaker and contents in a water bath maintained at 24.5 ± 0.3°C. and allow to stand for 45 min. Add 40 ml. of distilled water and mix the contents thoroughly. Pour approximately 10 ml. of the unsettled mixture into an empty filtering crucible having a bottom containing fine perforations, and allow the solution to filter without the use of suction. Apply suction very gently and filter the remainder of the solution, allowing the alpha-cellulose fibers to form the filtering medium. Pass the filtrate back through the crucible, and repeat this procedure until a filtrate free from alpha-cellulose fibers is obtained. Wash the alpha-cellulose residue with 250 ml. of distilled water at room temperature, rinsing out the beaker with the wash water. Transfer the combined filtrate and washings to an 800-ml. beaker, and save for the beta-cellulose determination. Add 20 ml. of 10% acetic acid solution to the crucible, and allow to pass through without the use of suction. Applying gentle suction, wash the alpha-cellulose mat with water at room temperature until the wash water does not react acid to litmus. Transfer the alpha-cellulose mat to the same weighing dish that was used in drying the original sample, and pull the fibers apart. Heat the dish and contents at 100 to 105°C. with the cover removed, replace the cover, cool in a sulphuric acid desiccator, weigh, and repeat until constant weight is obtained. Calculate the increase in weight of the weighing dish to percentage of alpha-cellulose in the dried sample.

### *Beta-Cellulose*

Add a few drops of phenolphthalein solution to the filtrate from the alpha-cellulose determination and acidify this with glacial acetic acid, adding 5 ml. excess. Heat the solution to 95°C. to coagulate the beta-cellulose, and allow the precipitate to settle. Prepare a filter as follows: flute a piece of Whatman no. 41 or similar 9.0-cm. filter paper and place in a weighing bottle equipped with a tight-fitting cover. Heat the bottle and contents at 100 to 105°C. for 2 hr. with cover removed. Replace the cover, cool in a sulphuric acid desiccator, and weigh. Place the fluted filter paper in a funnel and decant as much as possible of the clear supernatant liquid through the filter. Transfer the precipitate to the filter and rinse the beaker with boiling water. Transfer the filter paper and precipitate back to the weighing bottle, heat the bottle and contents at 100 to 105°C. with the cover removed, replace the cover, cool in a sulphuric acid desiccator, weigh, and repeat this procedure until constant weight is obtained. Calculate the increase in weight of the bottle to percentage of beta-cellulose in the dried sample. In the above determination use such filter paper that the gain or loss in weight of the paper, when a blank determination is run, is not more than 0.001 gram and when necessary make a suitable correction for this.

### *Gamma-Cellulose*

Calculate the percentage of gamma-cellulose by subtracting from 100 the sum of the percentages of alpha-cellulose and beta-cellulose.

## APPENDIX B

Chemical requirements: alpha-cellulose 90.00%, beta-cellulose 4.00%, and gamma-cellulose 6.00%.

### *Alpha-Cellulose*

Prepare a portion of the sample by shredding or fluffing. Transfer approximately 1 gram of the material to a tared weighing dish approximately 3 in. in diameter and equipped with a tight fitting cover, heat at 100 to 105°C., for 2 hr. with the cover removed. Replace the cover, cool in a sulphuric acid desiccator, and weigh. Carefully transfer the weighed sample to a 250-ml. beaker, add 25 ml. of a carbonate-free 17.5% sodium hydroxide solution at 24.5 ± 0.3°C. Place the beaker and contents in a water bath maintained at 24.5 ± 0.3°C. and allow to stand for 45 min. Add 40 ml. of distilled water and mix the contents thoroughly. Pour the unsettled mixture into a weighed coarse sintered glass crucible and allow the solution to filter with use of suction. Wash the alpha-cellulose residue with 250 ml. of distilled water, rinsing out the filtering flask with the wash water. Transfer the combined filtrate and washings to an 800-ml. beaker, and save for the beta-cellulose determinations. Add 20 ml. of 10% acetic acid solution to the crucible, and allow to pass through with the use of suction. Applying gentle suction, wash the alpha-cellulose mat with water at room temperature until the wash water does not react acid to litmus. Transfer the weighed coarse sintered glass crucible back to the same weighing dish that was used in drying the original sample. Heat the dish and contents at 100 to 105°C. with the cover removed, replace the cover, cool in a sulphuric acid desiccator, weigh, and repeat until constant weight is obtained. Calculate the increase in weight of the weighing dish to percentage of alpha-cellulose in the dried sample.

### *Beta-Cellulose*

Add a few drops of phenolphthalein solution to the filtrate from the alpha-cellulose determination and acidify this with glacial acetic acid, 20 ml. of acetic acid with agitation of solution. Heat the solution to 95 ± 5°C. to coagulate the beta-cellulose, and allow the precipitate to settle and filter hot (80 to 90°C.).

Prepare a filter as follows: place a 2.1-cm. no. 42 Whatman filter paper in the bottom of a no. 4 Gooch crucible. Put approximately 1 gram of fiberglas on top of the filter paper in the crucible. Place a Witt plate on the fiberglas mat and pour a small quantity of hot water through the filter Put filter in weighing bottle and heat at 100 to 105°C. for 2 hr. with cover removed. Replace the cover, cool in a sulphuric acid desiccator, and weigh. Decant as much as possible of the clear supernatant liquid through the filter. Transfer the precipitate to the filter and rinse the beaker with boiling water. Transfer the filter and precipitate back to the weighing bottle heat the bottle and contents at 100 to 105°C. with the cover removed, replace the cover, cool in a sulphuric acid desiccator, weigh, and repeat this procedure until constant weight is obtained. Calculate the increase in weight of the bottle to percentage of beta-cellulose in the dried sample.

### *Gamma-Cellulose*

Calculate the percentage of gamma-cellulose by subtracting from 100 the sum of the percentages of alpha-cellulose and beta-cellulose.


This work was part of a broad program carried out in the laboratories of the Scott Paper Co. at Everett, Wash. The writer wishes to acknowledge the helpful suggestions and assistance of his associates in connection with this study and in checking the new method under routine operating conditions.

# Variables Affecting Brightness of Unbleached Sulphite Pulp

DAVID L. MOSHER

Uniform raw materials are required for good operation of all mills. Physical properties such as strength and dirt content are examined frequently. Brightness, however, of unbleached sulphite is usually overlooked. Brightness is particularly important to mills that blend unbleached sulphite with bleached stocks to maintain a brightness level. A uniform blend is not possible with a brightness range of 15 points found during this project. It was decided to trace as many variables affecting brightness of unbleached sulphite pulp during the cooking process as possible. The following variables were selected: (1) percentage reducible sulphur present in the unbleached sulphite pulp, (2) percentage sulphur trioxide, (3) lignin content, (4) temperature, (5) composition of cooking acid, and (6) water purity.

## PERCENTAGE REDUCIBLE SULPHUR

Reducible or free sulphur may be introduced into the unbleached sulphite in two ways: (1) pulling sulphur during burner operation and (2) decomposition of the cooking liquor during cooking. Percentage reducible sulphur present in the unbleached sulphite was determined by TAPPI Standard T 406 M-46. Cooks were selected over a wide brightness range. A great number of cooks were analyzed and the percentage reducible sulphur correlated with the brightness on a graph. This correlation is shown on Fig. 1. The data shown in Fig. 1 support the theory advanced by Sutermeister (*1*) that reducible sulphur is formed by decomposition of the cooking liquor according to the following reaction:

$$3SO_2 \rightarrow 2SO_3 + S$$

It has been authoritatively stated that reducible sulphur acts catalytically and results in the formation of $H_2SO_4$ which combines with the lime to form $CaSO_4$ which reduces the amount of base present. In the absence of sufficient base the sulphonic acids would not be neutralized. Logically, when this occurs the brightness of the unbleached sulphite pulp is reduced. If this reaction continues at a sufficient rate due to excessive temperature, we can understand why the brightness would be lowered so markedly in the darker cooks. The brighter cooks show a range in brightness of (63 to 58) and were found to contain less than 0.01% of reducible sulphur. In this group of cooks the reducible sulphur range was (0.002 to 0.01%). The dark cooks show a range in brightness of (58 to 53) and were found to contain more than 0.01% reducible sulphur. In this group of cooks the reducible sulphur range was (0.01 to 0.06%).

## PERCENTAGE SULPHUR TRIOXIDE

The percentage of sulphur trioxide was determined by gravimetric analysis of the cooking liquor from the tower and accumulator. A West Coast authority gave us the following limits for a hot acid system as a guide: 0.055% $SO_3$ maximum present in the tower acid, and 0.075% $SO_3$ maximum present in the accumulator. Good operation of a hot acid system should not have more than 0.02% build-up between the tower acid and cooking liquor from the accumulator. Figure 2 shows that we were over the limits both in tower operation and accumulator. The most significant fact shown on Fig. 2 is the large percentage of sulphur trioxide being formed in the digester during the period of investigation. Samples were taken from the tower and accumulator for 18 days during October, 1953. We were experiencing a very wide range of brightness at this time (49.0 to 63). The amount of sulphur

David L. Mosher, Project Chemist, Fibreboard Products, Inc., Port Angeles, Wash.

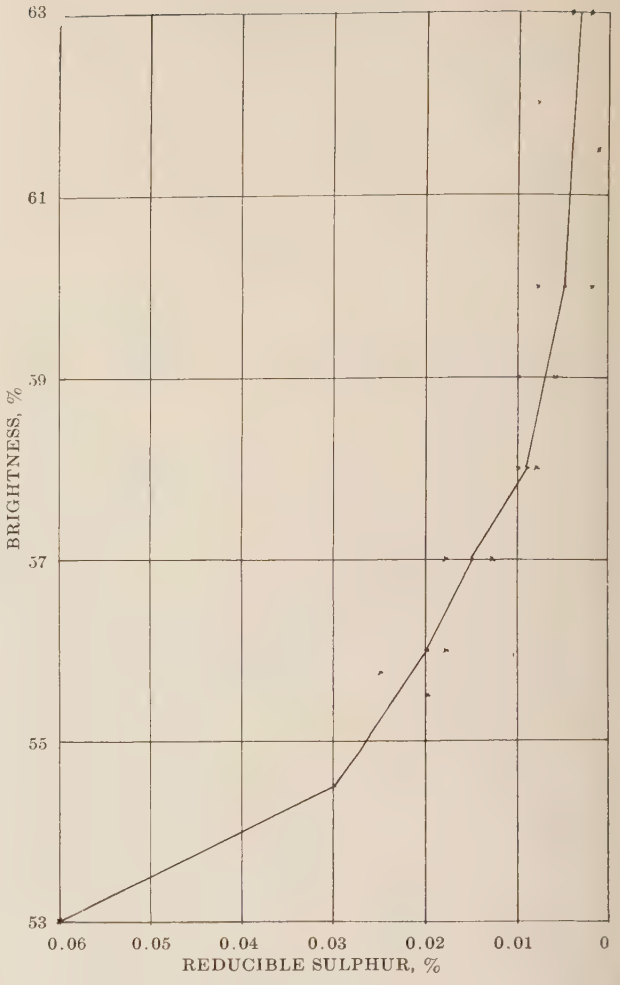



*Fig. 1. Per cent reducible sulphur in unbleached sulphite*

trioxide forming within the digester indicates to what extent the before-mentioned reaction, $3SO_2 \rightarrow 2SO_3 + S$ is taking place. The percentage of sulphur trioxide could not be correlated with the brightness in any one particular cook. However, it is felt that the large build-up between tower acid and cooking liquor during this period indicates to what extent the cooking liquor decomposed during the cooking cycle.

## LIGNIN CONTENT

The percentage of lignin remaining in the unbleached sulphite was suspected of lowering the brightness. TAPPI Standard T 222 m-52 for determination of lignin in wood pulp was chosen. TAPPI Standard T 6 m-50 for extracting the resin was also used. The investigation showed that we had an average of 0.6579% resins based on weight of moisture-free unbleached sulphite. It was found that the percentage lignin followed a definite trend, bright cooks having the least amount of lignin present. In one sense the percentage of

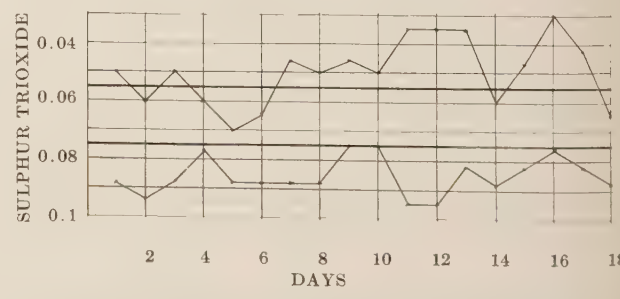



*Fig. 2. Per cent sulphur trioxide tower acid versus accumulator acid (heavy line—limits of build-up)*

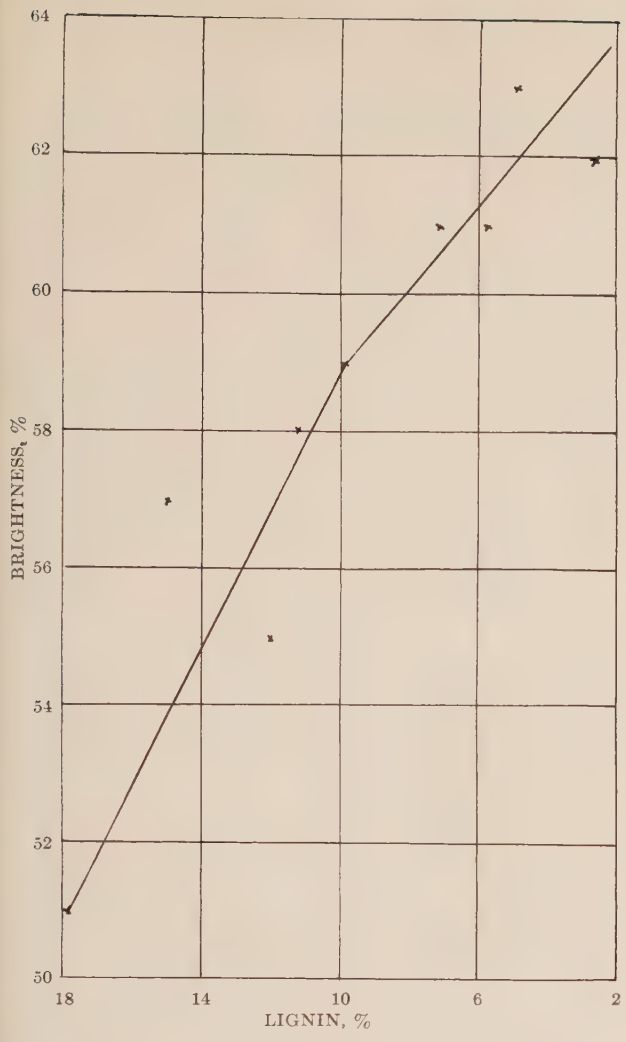



*Fig. 3. Per cent lignin in unbleached sulphite*

lignin remaining in any one cook is an indication of the amount of cooking or delignification undergone by that cook. Figure 3 illustrates the relationship of lignin and brightness of unbleached sulphite. The brighter cooks (63 to 58) contained from (2.3 to 11.1%) lignin. The darker cooks (58 to 52) analyzed from (11.1 to 17.8%). The lignin content follows the permanganate number indicating the degree a cook was delignified. In general, hard uncooked unbleached sulphite pulp is dark.

Literature to be cited concerning lignin content of unbleached sulphite and its effect upon brightness seemed to be limited. The author was referred, however, to a paper written by Kimble (*2*) which concerned the effect of lignin upon alkaline pulp color. The object of his work was to determine the source, relative importance, rate of development, and behavior of the colored materials in soda and kraft black liquors. He stated that the most important colored constituent is lignin, which sometimes accounts for 90% of the color. The color of kraft pulps is partially dependent on the coloring materials in the black liquor. It is unlikely that much of the color comes from the true cellulose portion because little darkening occurs when alpha-cellulose is cooked with kraft white liquor. The lignin seems to act upon unbleached sulphite in a similar manner according to this investigation.

## TEMPERATURE

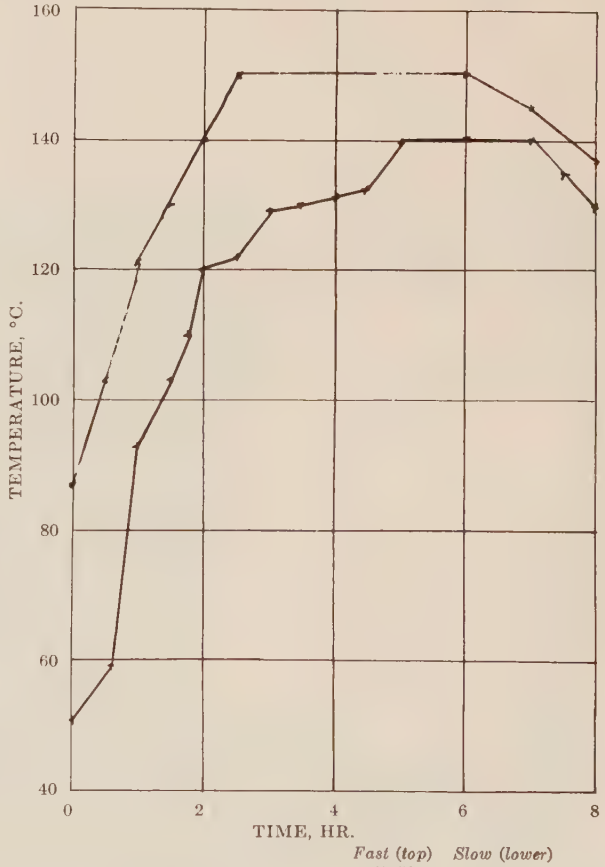
In order to illustrate the effect of a fast temperature rise as compared to a slow temperature rise, Fig. 4 was developed. It has been stated that the lowest temperature which gives a satisfactory degree of purity in a reasonable time is the best temperature to use. The empirical rule in kinetics which states that the rate of reaction is approximately doubled for each 10°C. rise in temperature can be applied to sulphite cooking. Figure 4 illustrates that temperature appeared to be a controlling factor concerning brightness. Unbleached sulphite exhibiting the best brightness properties was cooked with a slow temperature rise allowing ample time for complete penetration of the chips with the free sulphur dioxide and the slower penetrating combined sulphur dioxide. One-half hour precirculation with hot acid before steaming and $1^1/_2$-hr. rise to 110°C. was believed to be sufficient with the hot acid system under 80-lb. digester pressure. Uniform temperature must be maintained at all times within the digester. The rate of reaction of a given acid strength is controlled by the temperature and pressure.

There seems to be three main types of burning in cooking unbleached sulphite: (1) chemical burning—caused by decomposition of the cooking liquor or lack of penetration of the chips, (2) physical burning—brought about by relieving the cooking liquor level below the chips allowing them to be attacked by steam, and (3) overcooking or carrying the cook beyond its normal end point.

## COMPOSITION OF COOKING ACID

We use a Barker system in our plant to make the raw acid, employing dolomite lime for the base consisting approximately of 35% MgO–57% CaO.

Free sulphur dioxide in the cooking liquor is the active cooking agent. Our mill maintains a 5.7% free $SO_2$. In-




| | *Fast (top) curve* | *Slow (lower) curve* |
|---|---|---|
| Acid composition | | |
| Total $SO_2$ | 6.60 | 6.80 |
| Free $SO_2$ | 5.32 | 5.52 |
| Combined $SO_2$ | 1.28 | 1.28 |
| Physical properties | | |
| Burst, % | 41.6 | 36.8 |
| Permanganate no. | 14.6 | 10.6 |
| Dirt count | 90 | 90.0 |
| Brightness | 56.0 | 61.0 |

*Fig. 4. Fast versus slow temperature rise*

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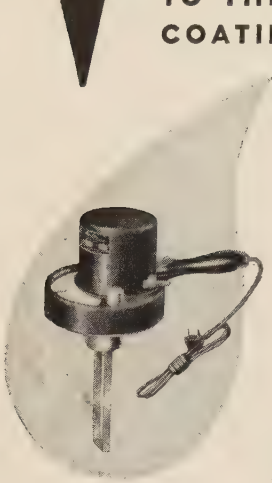

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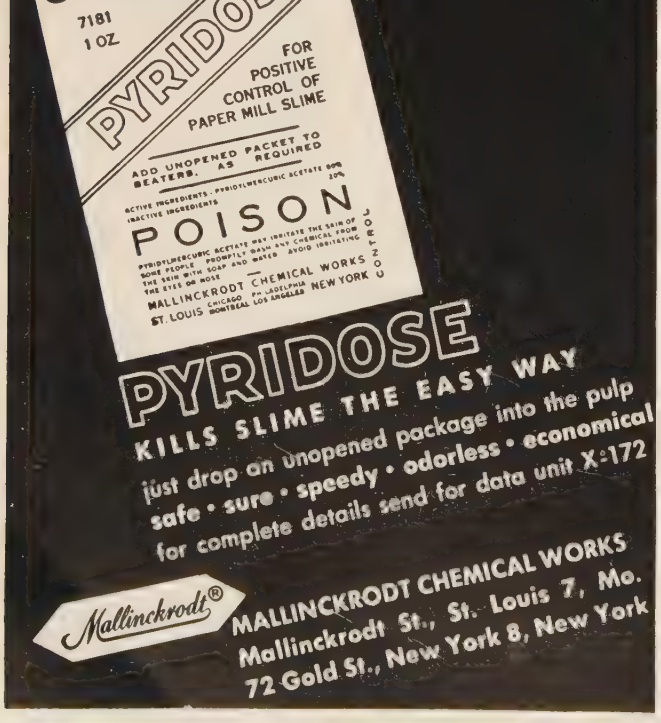
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creasing the percentage of free sulphur dioxide allows a reduction in temperature for a given cooking time.

As already indicated the main mechanism of sulphite pulping is one of sulphonation brought about by the action of free sulphur dioxide. The combined sulphur dioxide plays no role in this reaction. Its main function is to neutralize the strong lignosulphonic acids. This allows the free sulphur dioxide to continue cooking at high temperature without deteriorating the brightness of unbleached sulphite. Casey (*3*) stated that if lignosulphonic acids are heated above a temperature of about 100 to 110°C in the absence of the base ion dark colored products are formed.

A research project conducted by Rosenfeld (*4*) of Crown Zellerbach Corp. Camas, Wash., concerning chip packing and combined sulphur dioxide showed the following effect on brightness. Dropping the percentage of combined sulphur dioxide based on moisture-free wood weight from 6.03 to 4.22% as a result of extreme chip packing, the brightness dropped from 60.7 to 56.2. The combined sulphur dioxide content was increased to equal a normal packed digester, 6.03%, and the brightness increased from 56.2 to 62.5. This serves to illustrate the role played by the combined sulphur dioxide. It prevents the polymerization of the strong lignosulphonic acids into insoluble dark colored products. Our mill maintains a 4.7 to 1 liquor to wood ratio. The combined sulphur dioxide of the cooking liquor averages 1.30%. This gives us a combined sulphur dioxide based on moisture-free wood of 6.1%. Assuming that we have very little chip packing we should have a sufficient excess of combined sulphur dioxide to neutralize the sulphonic acids during the cook.

## WATER PURITY

We all know that pulp can be dyed. The presence of iron and organic materials effect the brightness in this way. We have had the occasion to observe in our plant that unbleached sulphite pulp brightness was effected when washed with water containing suspended materials. Normally this is not a factor concerning us for we have an unusually good water supply.

## CONCLUSION

It is concluded that percentage of reducible sulphur, percentage of sulphur trioxide, and percentage of lignin found to be present in the unbleached sulphite are linked together and controlled by temperature. It is also concluded that the presence of the above in the unbleached sulphite, beyond limits, is detrimental to the brightness.

It was found that the cooking acid will decompose forming reducible sulphur to some extent at all times. The free sulphur must be kept below 0.01% in order to realize 58.0 or better brightness. The formation of sulphur trioxide which is related to the formation of reducible sulphur according to the reaction

$$3SO_2 \rightarrow 2SO_3 + S$$

must be kept within limits. The composition of the cooking liquor must be maintained at a high percentage of free sulphur dioxide with a sufficient excess of combined sulphur dioxide present to neutralize the strong lignosulphonic acids given off during the cooking cycle.

Furthermore, it was concluded that ample time must be allowed for complete penetration of the chips in a digester before the critical temperature is attained (110°C.). A slow uniform cooking temperature proved to be our answer to bright unbleached sulphite pulp.

## LITERATURE CITED

1. Sutermeister, E., "Chemistry of Pulp and Paper Making," page 195, Third edition (1941).
2. Kimble, Glenn C. (Abstract), *Tech. Assoc. Papers* 42: 341 (1941).
3. Casey, James P., "Pulp and Paper," vol. 1, page 98 (1952).
4. Rosenfeld, Alan S., *Tappi* 10: 128A (Oct., 1953).

# UNITED STATES PATENTS ON PAPERMAKING

*Second Quarter, 1954* — *Compiled by W. B. Weber*

THE following list of United States patents has been compiled from the current numbers of the Official Gazette of the United States Patent Office. Because, as a rule, only one claim is published in the Gazette, it is not claimed that the list is complete; also, it is possible that the list may contain some patents that do not apply specifically to pulp and paper manufacture. Copies of any of the following patents may be obtained from the United States Patent Office, Washington 25, D. C., by sending twenty-five cents for each patent desired.

### April 6, 1954

Bruker, Hobart W. Web corrugating machine. U. S. patent 2,674,299. Filed March 20, 1952. 8 claims. Assigned to George W. Swift, Jr., Inc. [Cl. 154-31.] Several adhesive-applying units, each of less width than the machine, may be adjusted to apply adhesive across the entire roll or only a portion thereof.

Burke, William F., and Essex, Robert L. Apparatus for imprinting carbon on paper. U. S. patent 2,674,225. Filed Aug. 10, 1951. 7 claims. Assigned to The McBee Co. [Cl. 118-202.] Both printing and impression cylinders are heated by electric elements to ensure a uniform coating of carbon wax on the paper.

Davidson, Bruce J. Display stand. U. S. patent 2,674,377. Filed Dec. 30, 1949. 3 claims. Assigned to National Biscuit Co. [Cl. 211-72.]

Ehmann, Leslie G. Side grip carton handling means for lift trucks. U. S. patent 2,674,387. Filed Aug. 10, 1950. 2 claims. Assigned to Hyster Co. [Cl. 214-653.] Opposed arms equipped with pressure plates coupled by an equalizer bar may be brought together to hold cartons between the plates.

Haug, Anton J. Machine for reducing and treating fibrous papermaking materials. U. S. patent 2,674,162. Filed Dec. 18, 1950. 23 claims. [Cl. 92-26.] Horizontal cylindrical casing within which a small diameter roll (also driven) operates and a screw conveyor adjacent one side wall acts to advance material to the effluent end of the refiner.

Hoefle, Milton L., and Wynn, Robert W. Fluorescent brighteners. U. S. patent 2,674,604. Filed Nov. 15, 1951. 4 claims. Assigned to General Aniline & Film Corp. [Cl. 260-346.2.]

Khndzorian-Iablokoff, Arthur, Delord, Andre, Falinski, Marie, and Lautie, Raymond. Process for treating wood and resulting article. U. S. patent 2,674,543. Filed March 4, 1949. 2 claims. Assigned to Office National d'Etudes et de Recherches Aeronautiques. [Cl. 117-57.] Wood in veneer form is delignified by soaking in sodium chlorite solution, then the intercellular voids filled with resin or other material under heat and pressure.

Leek, Thomas G. Pigment comprising resin coated clay particles. U. S. patent 2,674,587. Filed Nov. 24, 1951. 5 claims. Assigned to Edgar Brothers Co. [Cl. 260-38.] Clay dispersion containing formaldehyde resin is spray-dried to form resin-coated particles wherein the resin is polymerized during drying of the sheet.

Messinger, William, and Volkmann, Joseph. Remote-control nip adjustment and sequence of nip closure control. U. S. patent 2,674,222. Filed June 6, 1950. 30 claims. Assigned to St. Regis Paper Co. [Cl. 118-10.] Closure of nips in coater rolls may be made automatically and in any desired sequence, then final adjustment of the nip made after closure; automatic opening is provided for in event of power or other failure.

Pahl, Walter L., Williams, Keith H., and Tierney, Hubert J. Gummed strapping tape. U. S. patent 2,674,555. Filed Aug. 17, 1950. 2 claims. Assigned to Minnesota Mining & Manufacturing Co. [Cl. 154-53.5.] Laminated tape contains a layer of rubber-resin adhesive in which are embedded rayon fiber yarns possessing high tensile strength and good elasticity.

Pahl, Walter H., Williams, Keith H., and Tierney, Hubert J. Gummed strapping tape. U. S. patent 2,674,556. Filed Aug. 17, 1950. 1 claim. Assigned to Minnesota Mining & Manufacturing Co. [Cl. 154-53.5]. Glass fiber yarn is embedded in an adhesive layer to produce tape with tensile strengths of more than 400 lb. per inch width.

Potts, Ralph H. Process for distilling tall oil. U. S. patent 2,674,570. Filed Oct. 17, 1950. 4 claims. Assigned to Armour and Co. [Cl. 202-52.] Tall oil is subjected to fractional distillation to separate the fatty acids, resin acids, etc.; the rosin acids are steam stripped from the pitch.

Prevost, Bruno E. Apparatus for controlling Jordan engine plugs. U. S. patent 2,674,163. Filed July 6, 1949. 3 claims. Assigned to John W. Bolton & Sons, Inc. [Cl. 92-27.] Shaft bearings for jordan plugs are mounted on raceways so that the bearings are moved as the plug is advanced to compensate for wear. The shaft is adjustably spring loaded to take up slack in the linkage.

Ross, Colin A. Shipping carton. U. S. patent 2,674,400. Filed Jan. 3, 1950. 2 claims. Assigned to Behr-Manning Corp. [Cl. 229-37.] Octagonal shipping carton for heavy rolled material.

Sams, Fred V. Hydraulic log barker with oscillating jet issuing means. U. S. patent 2,674,283. Filed April 3, 1950. 6 claims. Assigned to Allis-Chalmers Manufacturing Co. [Cl. 144-208.] Jet nozzles are mounted to oscillate in a path transverse to the long axis of the log to make the streams most effective.

Schweich, Edward F. Container. U. S. patent 2,674,372. Filed June 14, 1950. 8 claims. Assigned to Lewin-Mathes Co. [Cl. 206-52.] Octagonal-shaped container for coils of tubing or the like.

Wallin, Sven W., and de Frumerie, Nils G. M. Regulating device. U. S. patent 2,674,178. Filed Oct. 17, 1951. 4 claims. Assigned to Aktiebolaget Svenska Fläktfabriken. [Cl. 98-38.] Interconnected valves (especially for air conditioning and the like) mix desired portions of air or gases with a minimum of noise.

### April 13, 1954

Anderson, Wilbur G., Jr. Bottom structure for reinforced carrier receptacles. U. S. patent 2,675,156. Filed Feb. 5, 1953. 3 claims. Assigned to American Box Board Co. [Cl. 229-23.] Case for bottled goods has a framework of metal rods and paperboard covering for the sides and bottom, the latter being easily replaceable.

Baird, Samuel P. Package of plurality of cartons and method of packaging. U. S. patent 2,675,123. Filed Oct. 20, 1949. 3 claims. [Cl. 206-65.] Eight three-sided corner sections are arranged around objects to form an adjustable shipping container fastened with tape, bands, adhesive, etc.

Buttery, Kenneth T. Knockdown covered carton. U. S. patent 2,675,160. Filed Jan. 9, 1951. 3 claims. Assigned to Sutherland Paper Co. [Cl. 229-33.] Clothing box with hinged cover has locking tongue on the closure flap which engages a slit in the front wall of the carton body.

Buttery, Kenneth T. Collapsible carton. U. S. patent 2,675,161. Filed Jan. 9, 1951. 8 claims. Assigned to Sutherland Paper Co. [Cl. 229-34]. Interior sloping walls of this tray support articles packaged therein and prevent displacement.

Buttery, Kenneth T. Covered carton or container. U. S. patent 2,675,163. Filed Jan. 27, 1950. 10 claims. Assigned to Sutherland Paper Co. [Cl. 229-36.] Lightweight paperboard carton with interlocked end walls, hinged cover, and closure flap is claimed to be rigid.

Carpenter, Robert J. Log-peeling machine having rockably mounted flail-carrying shaft. U. S. patent 2,675,035. Filed Feb. 5, 1953. 4 claims. [Cl. 144-208.] Flail-bearing shaft is supported between independently movable levers that are free to ride over irregularities in the logs.

Clark, John D. Combined cover and handle structure for paperboard boxes. U. S. patent 2,675,168. Filed Jan. 18, 1950. 3 claims. Assigned to The Hinde & Dauch Paper Co. [Cl. 229-44.] Extensions of the end walls form handles and protrude through the cover flap which locks them in place.

Clemens, Ludwig. Endless band pressing machine for manufacturing upholstered paper. U. S. patent 2,675,053. Filed May 12, 1949. 4 claims. [Cl. 154-30.] Multiple layers are embossed with a pattern by passing the webs between endless belts comprising pressure plates containing heating elements.

Coghill, James T. Machine for treating wood pulp. U. S. patent 2,674,928. Filed Dec. 30, 1948. 9 claims. Assigned to

Curlator Corp. [Cl. 92-26.] Refiner comprises stationary working surface opposed by upper surface which oscillates in a gyratory motion. Surfaces are roughened to provide traction on the pulp.

Deline, Edwin F., and Plack, Roy M. Bottle case partition. U. S. patent 2,675,167. Filed Oct. 27, 1950. 1 claim. [Cl. 229-42.] Partition strips cut in a zig-zag pattern and assembled in a case tend to guide misaligned bottles into the proper cell.

Gray, Kenneth R., Crosby, Hartzell L., and Steinberg, John C. Solution phase process of converting sodium sulphide into sodium salts of carbonic acid. U. S. patent 2,675,297. Filed June 7, 1950. 7 claims. Assigned to Rayonier Inc. [Cl. 23-63.] Carbonation of sulphide-containing liquors by stages involves carbonation under pressure and steam stripping of hydrogen sulfide at reduced pressure.

Green, Barrett, K. Heat sensitive record material. U. S. patent 2,675,332. Filed Jan. 20, 1951. 2 claims. Assigned to The National Cash Register Co. [Cl. 117–69.] Paper treated with an eosin compound leaves a red impression where a heated marking or printing element comes into contact with such paper.

Gwinn, Myron B., and Hopkin, Donald E. Apparatus for forming spots of adhesive on a moving web. U. S. patent 2,674,974. Filed March 16, 1953. 6 claims. [Cl. 118-210.] Grooved applicator roll is opposed by a drum fitted with rubber tabs which force the web into contact with the applicator roll.

Haas, Richard G. Knockdown covered carton. U. S. patent 2,675,164. Filed Dec. 22, 1950. 4 claims. Assigned to Sutherland Paper Co. [Cl. 229-36.] Extensions of the front and rear walls engage slits in the end walls to make the carton easily erected or knocked down.

Higham, Walter E. Basket and the like made from cardboard or similar sheet materials. U. S. patent 2,675,162. Filed Feb. 17, 1953. 9 claims. Assigned to J. Dring Ltd. [Cl. 229-34.] Double-walled basket folded from a single blank contains sidewalls perforated for ventilation.

Howland, Louis H. Preparation of synthetic rubber-carbon black mixture using the residue from the alkali extraction of bark. U. S. patent 2,675,361. Filed March 14, 1952. 6 claims. Assigned to United States Rubber Co. [Cl. 260-17.4.] Alkali-extracted comminuted bark from Douglas-fir, redwood, or gumwood is added to synthetic rubber latex as an extender.

Lammers, Gerald J. Loaded paper. U. S. patent 2,675,312. Filed Sept. 29, 1950. 1 claim. [Cl. 92-21.] Reclaimed paper utilizing kraft paper and as high as 40% newsprint contains about 2% of a sulphur composition as a sizing and hardening agent.

Layte, Ralph R. Oil filter element construction. U. S. patent 2,675,127. Filed June 14, 1951. 3 claims. Assigned to Purolator Products, Inc. [Cl. 210-169.] Filter element composed of a pleated layer of laminated filter papers arranged in order of decreasing porosity toward the downstream side of the filter.

Long, John C. Coil holder. U. S. patent 2,675,171. Filed Jan. 2, 1951. 1 claim. [Cl. 229-92.9.]

Main, James W. Paperboard container. U. S. patent 2,675,-166. Filed July 9, 1952. 4 claims. Assigned to Container Corporation of America. [Cl. 229-38.] Container for the shipment of fruit or the like is folded from a single cardboard blank and comprises double side and end walls with the end wall slightly higher to provide ventilation when the cases are stacked.

Natwick, John W. Paper pulp process and apparatus. U. S. patent 2,675,311. Filed Aug. 9, 1948. 15 claims. [Cl. 92-6.] A series of digesters are connected so that chips and liquor are pumped from the bottom of one to the top of the next, the pressure and temperature increasing progressively to the last digester from which the pulp is withdrawn and separated from the liquor. Vapors and noncondensable gases are constantly removed from the heads of the digesters and from the liquor heaters attached to each digester, thus increasing the concentration of the liquors as they progress through the system.

Parker, Matthew, and Harris, Richard A. Feeding of wrapping materials to wrapping machines and the like. U. S. patent 2,675,230. Filed Feb. 4, 1952. 7 claims. Assigned to The Forgrove Machinery Co., Ltd. [Cl. 271-2.6.] Underfeeding of the wrapping sheet stops this machine until the feed mechanism is restored to normal.

Reiner, George. Egg container. U. S. patent 2,675,159. Filed Nov. 7, 1951. 4 claims. [Cl. 229-28.] Paperboard egg carton.

Ringler, William A. Reinforced partitioned carrier. U. S. patent 2,675,158. Filed Feb. 23, 1950. 9 claims. Assigned to The Gardner Board and Carton Co. [Cl. 229-28.] Bottle carrier.

Roshkind, Allan I., Stephenson, Paul A., Rau, Eric J., and Foster, Thomas W. Alternative opening carton. U. S. patent 2,675,165. Filed Nov. 2, 1950. 3 claims. Assigned to Container Corporation of America. [Cl. 229-38.] Flat paperboard carton for sheet material has tuck flaps on one side and one end to provide a choice of access.

Schwartzberg, Louis. Egg shipping container. U. S. patent 2,675,319. Filed March 21, 1950. 1 claim. [Cl. 99-177.] Molded pulp separator sheets and cardboard partition structure prevent breakage.

Sebesta, Alfred C. Record card construction. U. S. patent 2,675,170. Filed Sept. 16, 1950. 1 claim. Assigned to International Business Machines Corp. [Cl. 229-68.] Two sheets of paper are cemented together to form an envelope with a punch card extension at one end for record purposes.

Tamarin, Bernard J. Cigarette package and method of making same. U. S. patent 2,675,169. Filed Sept. 23, 1948. 19 claims. Assigned to Pull-Packaging, Inc. [Cl. 229-51.] Tear strip attached to inner wrapping of cigarette package opens an end of the folded wrapper before detaching completely.

Theobald, Delbert A. Control means for web slitting and the like. U. S. patent 2,675,437. Filed Dec. 22, 1950. 7 claims. Assigned to Container Corporation of America. [Cl. 200-61.18]. Feeler element in contact with the edge of the web controls slitter wheel to compensate for lateral shifting and keeps edge trim to a minimum.

Turner, William E., and Foster, Thomas W. Retractable handle bottle carrier. U. S. patent 2,675,157. Filed April 12, 1948. 5 claims. Assigned 50% to Container Corporation of America and 50% to O. B. Andrews Co. [Cl. 229-28.] Bottle carrier with wire handle.

Vander Lugt, Thomas, Jr. Bottle carrier. U. S. patent 2,675,264. Filed June 23, 1951. 10 claims. Assigned to Sutherland Paper Co. [Cl. 294-87.2.] The tops of two gabled-top milk cartons are inserted into this carrier and fold-in side flaps engage the tops of the cartons.

Wicksell, Sture V. Disintegrator for fibrous materials. U. S. patent 2,674,927. Filed Jan. 27, 1951. 7 claims. [Cl. 92-23.] Disintegrating rotor, strainer plate, and pump wheel rotate as a unit at the bottom of the tub to disintegrate pulp bundles, waste paper, etc. and continuously remove accepted stock from the unit.

Wittkuhns, Bruno A., Nutting, H. George D., and Hill, Gilbert F. Machine for preparing paper container blank]. U. S. patent 2,674,929. Filed April 6, 1949. 14 claims. Assigned to National Paper Bottle Co., Inc. [Cl. 93-36.] Machine for forming milk cartons having cylindrical base sections and truncated conical tops; the carton is fillable on the same machinery as that used for glass bottles.

**April 20, 1954**

Constance, Joseph A. Method of coating paper. U. S. patent 2,676,119. Filed March 13, 1953. 7 claims. Assigned to Brightwater Paper Co. and Millers Falls Paper Co. Casein or gum arabic size with pigment is applied to typewriter paper to control ink penetration.

Conti, John D. Wrapper making machine. U. S. patent 2,675,746. Filed Feb. 18, 1949. 6 claims. Assigned to American Viscose Corp. [Cl. 93-1.] Wrapping material is cut into lengths with a band of adhesive tape applied one half to the edge of said wrapping material and the other half exposed for securing the wrapped package.

Currie, Grover C. Bottle carrier. U. S. patent 2,675,956. Filed Feb. 19, 1949. 1 claim. Assigned to Dacam Corp. [Cl. 229-37.]

Foster, Thomas W. Tableware tray and insert. U. S. patent 2,675,912. Filed Aug. 19, 1949. 3 claims. Assigned to Container Corporation of America. [Cl. 206-45.19.] Insert is slotted with ridged slots to hold individual items of tableware.

Fuller, James M., and Walton, Thomas H. Process of preparing plastic substances and mold compositions and products thereof. U. S. patent 2,676,108. Filed Sept. 14, 1949. 14 claims. Assigned to Masonite Corp. [Cl. 106-38.5.] Water solubles from board manufacture are concentrated and heated with a polycarboxylic acid to form a film substance or binder for sand foundry cores.

Gibbons, Clyde F. Furniture container. U. S. patent 2,675,-955. Filed Oct. 22, 1949. 1 claim. Assigned to Gaylord Container Corp. [Cl. 229-23.] Raised portion of the cover accommodates the back of chairs, divans, etc.

Greiner, Charles J., and Sabee, Reinhardt N. Apparatus for segregating stacks of sheet material from a continuously forming supply of the material. U. S. patent 2,675,747. Filed Jan. 13, 1950. 18 claims. Assigned to International Cellucotton Products Co. [Cl. 93-93.] Interfolded web of tissue is passed between rolls where the web is folded and stacked in groups of the desired number of sheets and the stacks continuously removed from the bottom of the pile.

Hanson, Wallace E. Foldable display box. U. S. patent 2,675,913. Filed Aug. 11, 1953. 4 claims. Assigned to Sample-Durick Co., Inc. [Cl. 206-45.21.] Arcuate score lines in the side walls aid in forming a display box for the contents.

Hughes, Walter J. Chemical feeder. U. S. patent, 2,675,758. Filed Jan. 6, 1949. 12 claims. Assigned to Infilco Inc. [Cl.

103-38.] Diaphragm pump contains a diaphragm molded to the chamber and actuated by fluid pressure on the reverse side of the diaphragm.

Johnstone, Henry F. Recovery of sulphur dioxide contained in waste gases. U. S. patent 2,676,090. Filed Jan. 26, 1953. 15 claims. Assigned to Texas Gulf Sulphur Co. [Cl. 23-178.] Sulphur dioxide gases are absorbed in an ammoniacal solution as ammonium sulphite compounds and then treated with sulphuric acid to release sulphur dioxide gases and recover ammonium sulphate from the residual liquor.

Kao, John Y. L. Process of coating fibers with gas agitation. U. S. patent 2,676,099. Filed Sept. 25, 1948. 5 claims. Assigned to F. D. Farnam Co. [Cl. 92-21.] Air or other gas is introduced to produce agitation of a dilute pulp slurry during addition of the coagulant and the latex or resinous emulsion.

Marotta, Ralph. Fire retardant coating compositions containing a reaction product of phosphoryl chloride and anhydrous ammonia and articles coated therewith. U. S. patent 2,676,162. Filed June 28, 1949. 18 claims. Assigned to Monsanto Chemical Co. [Cl. 260-38.] Film claimed to be water and weather resistant forms a coating for paper, wood, etc. that intumesces when exposed to flame or heat.

Novak, Izador J. Protection of dispersed asbestos fibers on papermaking machines. U. S. patent 2,675,745. Filed Sept. 10, 1949. 5 claims. Assigned to Raybestos-Manhattan, Inc. [Cl. 92-38.] Chrysotile asbestos is defiberized in an alkaline dispersing agent and the sheet formed from this slurry.

Patterson, George H., and Balon, Walter J. Water-soluble derivatives of unsulphonated lignin. U. S. Patent 2,676,170. Filed July 15, 1950. 4 claims. Assigned to E. I. du Pont de Nemours & Co. [Cl. 260-124.] Alkali lignin is condensed with formaldehyde and chloracetic acid to produce a material valuable as a tanning agent.

Thurston, Benjamin L. Display box. U. S. patent 2,675,911. Filed March 27, 1952. 3 claims. Assigned to Frost Box Co., Inc. [Cl. 206-45.13.] False bottom is elevated by means of a bracket attached to the cover when the cover is opened.

Vogel, Frank W. Drinking cup. U. S. patent 2,675,954. Filed March 3, 1952. 1 claim. [Cl. 229-14.] Double-walled paper cup for improved insulating properties.

Vogt, Clarence W. Package for plastic commodities. U. S. patent 2,676,106. Filed June 2, 1950. 2 claims. [Cl. 99-171.] Flexible material is reinforced with a stiff material on three sides and the whole packaged inside an outer carton.

Wagner, Ernst. Process for the preparation of chlorine dioxide. U. S. patent 2,676,089. Filed June 6, 1949. 3 claims. Assigned to Deutsche Gold- und Silber-Scheideanstalt vormals Roessler. [Cl. 23-152.] Sodium chlorate solution is activated with dilute sulphuric acid in the presence of a foaming and reducing agent and the foam introduced into the top of a packed tower. The foaming and reducing agent is claimed to control the evolution of gas from the chlorate solution.

Youngfelt, Thomas T. Handling system for boardlike articles. U. S. patent 2,675,929. Filed June 28, 1946. 9 claims. Assigned to United States Gypsum Co. [Cl. 214-6.] Conveyor system for transporting boards from a drier to the stacking point and inverting one board so that face side of all boards is toward the inside of the stack.

Youngfelt, Thomas T., and Besal, Joseph A. Apparatus for forming stacks of boards. U. S. patent 2,675,930. Filed June 28, 1946. 11 claims. Assigned to United States Gypsum Co. [Cl. 214-6.] Very similar to U. S. patent 2,675,929. See abstract above.

Zimmerman, Donald R. Bag closure. U. S. patent 2,675,957. Filed Aug. 5, 1952. 3 claims. Assigned to Marathon Corp. [Cl. 229-62.] Paper folded in an "M" shape with adhesive-coated side inward is inserted in the bag mouth to provide an air-tight seal.

### April 27, 1954

Argodale, John. Paperboard container. U. S. patent 2,676,749. Filed May 26, 1951. 2 claims. Assigned to Twin Cities Container Corp. [Cl. 229-34.] Open-topped basket with wire handles that may be extended for carrying or retracted for stacking.

Baker, Arthur W., and Porter, George G. Cell case partition assembling machine. U. S. patent 2,676,522. Filed May 7, 1949. 11 claims. Assigned to The J. H. Mayhew Co. [Cl. 93-38.]

Baselt, Frederick C. Container cap with foldable barrier. U. S. patent 2,676,744. Filed Aug. 10, 1950. 5 claims. Assigned to American Can Co. [Cl. 229-7.] Friction plug closure for paperboard milk carton incorporates barrier webs at either side which are erected upon opening to prevent entry of foreign materials.

Blumberg, Samuel. Writing pad. U. S. patent 2,676,818. Filed Sept. 18, 1952. 3 claims. [Cl. 282-22.] Pad for use in the dark incorporates a plastic sheet with phosphorescent agent added placed over a carbon transfer sheet. Writing on the plastic sheet causes transfer of carbon onto paper pad.

Bolding, Hubert V. Container closure lock. U. S. patent 2,676,751. Filed April 9, 1951. 1 claim. Assigned to The Bradley & Gilbert Co. [Cl. 229-39.] Locking tongue on the bottom flap engages a corresponding flap on the side wall to form an effective lock.

Brook, Leonard. Wrapping machine. U. S. patent 2,676,441. Filed April 21, 1952. 9 claims. Assigned to The Forgrove Machinery Co. Ltd. [Cl. 53-41.] Small articles such as candies may be bunched or twist wrapped by this machine.

Friedman, Samuel. Jewelry shipping carton and display card. U. S. patent 2,676,699. Filed Feb. 28, 1952. 3 claims. [Cl. 206-45.19.] The carton is erected for shipping or flattened for display purposes.

Gastright, Frank A. Flat folded carton. U. S. patent 2,676,750. Filed March 15, 1951. 4 claims. Assigned to The Queens City Manufacturing Co. [Cl. 229-39.] Cut-out portion at the top facilitates entry of the opener blade and positive erection of the carton.

Geisler, William. Shipping case. U. S. patent 2,676,745. Filed Sept. 12, 1949. 2 claims. Assigned to Wilbro Corp. [Cl. 229-16.] Fold lines of a corrugated shipping case are reinforced by the application of a band of latex-sodium silicate composition about 1 in. wide along each fold line.

Goddard, Harold O. Waste sulphite liquor recovery. U. S. patent 2,676,883. Filed April 2, 1949. 9 claims. [Cl. 92-2.] An integrated system of handling blow-down gases and spent liquor is claimed in which maximum recovery of heat at each stage is the objective. Production of alcohol by fermentation of liquor and drying of concentrated liquor is described. Countercurrent washing of the pulp to recover maximum solids is one of the features of the patent.

Hamburg, Herman. Manufacture of articles such as boards and sheets from fibrous vegetable materials. U. S. patent 2,676,885. Filed Sept. 6, 1951. 3 claims. Assigned to Syntics Ltd. [Cl. 92-21.] Pulp from sawdust, bagasse, peat, etc. is produced by digestion with caustic soda, washing, precipitation of an insoluble sulphate within the pulp and the article formed under heat and pressure.

Hamburg, Herman. Manufacture of articles such as boards and sheets from fibrous vegetable materials. U. S. patent 2,676,884. Filed Dec. 22, 1948. 3 claims. Assigned to Syntics Ltd. [Cl. 92-21.] Very similar to U. S. patent 2,676,885, with the addition of passing the digested material between rolls to aid in defibering.

Kellogg, Richard A., and Martin, Richard H. Feeder carton. U. S. patent 2,676,746. Filed Feb. 16, 1951. 1 claim. Assigned to The Lawrence Paper Co. [Cl. 229-17.] Score line along the side of a carton makes possible the extension of a flap to form a feed trough.

Klein, Clarence F. Collapsible paperboard package and carrier. U. S. patent 2,676,731. Filed Aug. 13, 1949. 2 claims. Assigned to Frankenberg Bros., Inc. [Cl. 220-113.] Interior packing device that becomes a carrier upon removal from the shipping container.

Kottmann, Arthur A. Folding and sealing mechanism for wrapping machines. U. S. patent 2,676,445. Filed Feb. 28, 1952. 12 claims. Assigned to American Machine & Foundry Co. [Cl. 53-149.] Thermoplastic-coated wrapper is folded about the article and the article passed through a sealing channel where the coating is fused.

Lange, Ferdinand. Folded box. U. S. patent 2,676,747. Filed Nov. 24, 1950. 2 claims. Assigned to Werner Bahlsen. [Cl. 229-31.] Folded pleats overlapping in alternate directions make the corners of this box.

Montgomery, William J., and Taylor, William P. Apparatus for coating paper. U. S. patent 2,676,563. Filed Feb. 5, 1949. 8 claims. Assigned to The Champion Paper and Fibre Co. [Cl. 118-227.] Coating is applied by metering rolls to a rubber-covered roll against which operates a small-diameter doctor roll with associated wiping means.

Piazze, Thomas E. Container filling, closing, and sealing machine. U. S. patent 2,676,443. Filed March 12, 1949. 10 claims. Assigned to Continental Can Co., Inc. [Cl. 53-144.] Bags are filled, closed, and heat-sealed.

Pollak, Arthur. Lignin reinforced rubber and method of making same. U. S. patent 2,676,931. Filed June 5, 1952. 6 claims. Assigned to West Virginia Pulp & Paper Co. [Cl. 260-17.5.] Acid-precipitated lignin from kraft black liquor is incorporated into butadiene rubber as a reinforcing agent along with carbon black.

Scully, Edwin N. Dispensing carton for package wrapping ribbons. U. S. patent 2,676,701. Filed Dec. 27, 1949. 3 claims. [Cl. 206-58.] Package contains a saw edge to sever the ribbon at the desired point.

Sprenger, Wilbur V., and Roche, John C. Compartmented tray. U. S. patent 2,676,748. Filed Aug. 17, 1949. 2 claims. Assigned to Chicago Carton Co. [Cl. 229-32.] Tray divided

into three compartments is overwrapped with transparent foil to complete the package.

Strecker, Gerald. Sheet counting apparatus. U. S. patent 2,676,523. Filed Feb. 21, 1951. 11 claims. Assigned 50% to Kommandit-Gesellschaft, Dr. Otto C. Strecker, and 50% to Maschinenfabrik zum Bruderhaus Gesellschaft mit beschränkter Haftung. [Cl. 93-93.] Adjustable gearing units make the device adaptable for various ream counts and the strip inserting mechanism is synchronized with the cutter for high-speed operation.

Trillich, Bertram L. Self-sealing wrapping material. U. S. patent 2,676,897. Filed June 20, 1950. 6 claims. Assigned to The Munising Paper Co. [Cl. 117-68.] Wax coating is applied to one side of a sheet and the other side coated with a cohesive, substantially nonadhesive resinous material.

Weber, Herbert H., and Donnelly, Daniel W. Cutting apparatus for paper bag machinery. U. S. patent 2,676,657. Filed Sept. 6, 1949. 8 claims. Assigned to H. G. Weber & Co., Inc. [Cl. 164-48.] Bag tubing is notched prior to cutting into bag lengths, said notches aiding in opening the finished bag.

### May 4, 1954

Backhouse, Headley T., and Tebbs, George W. Sheet feeding apparatus. U. S. patent 2,677,542. Filed Nov. 9, 1949. 9 claims. Tebb's interest assigned to Backhouse. [Cl. 271-8.] Pusher wheels driven from the running-in wheels engage the rear edge of the sheet to force it against the front lay of the layboard.

Burger, Martin. Egg carton. U. S. patent 2,677,491. Filed May 11, 1948. 7 claims. Assigned to Shellmar Products Corp. [Cl. 229-28.] Paperboard egg carton.

Buttery, Kenneth T. Covered box. U. S. patent 2,677,492. Filed July 17, 1950. 4 claims. Assigned to Sutherland Paper Co. [Cl. 229-31.] Collapsible covered carton with the rear wall acting as a hinge between the top and bottom sections.

Buttery, Kenneth T. Collapsible automatic setup carton. U. S. patent 2,677,494. Filed Oct. 11, 1950. 3 claims. Assigned to Sutherland Paper Co. [Cl. 229-39.] Interlocking flaps in the bottom construction engage upon application of pressure at opposite corners, thereby erecting the carton.

Buttery, Kenneth T. Automatic setup carton. U. S. patent 2,677,495. Filed May 11, 1953. 4 claims. Assigned to Sutherland Paper Co. [Cl. 229-39.]

Crane, Harold S. Molded pulp carton. U. S. patent 2,677,490. Filed July 23, 1948. 13 claims. Assigned to Shellmar Products Corp. [Cl. 229-15.] Molded pulp egg carton.

Currie, Grover C., Dans, Bernard D., Wilkens, Louis F., and Dunn, Maurice B. Machine for packaging cylindrical articles. Filed Dec. 26, 1951. 95 claims. Assigned to Dacam Corp. [Cl. 53-34.] Automatic machine for packaging cans in a carrier, then placing groups of carriers into a shipping container.

Dana, Homer J., and Albrook, Raymond L. Sheet for electrical inscription. U. S. patent 2,677,651. Filed Oct. 9, 1950. 11 claims. Assigned to State College of Washington. [Cl. 204-2.] Base sheet for facsimile transmission is impregnated with a stable nitrate salt and an organic compound with a B-naphthyl amine radical which changes color when an electric current is passed through the sheet.

Ebert, Ludwig. Rearwardly slanting collapsible cardboard floor stand. U. S. patent 2,677,469. Filed Dec. 9, 1950. 2 claims. Assigned to Einson-Freeman Co., Inc. [Cl. 211-73.]

Fischer, Paul E., and Paulsen, Neal I. Method and mechanism for folding carton flaps. U. S. patent 2,677,220. Filed Dec. 23, 1950. 9 claims. Fischer's interest assigned to General Mills, Inc. and Pulsen's interest assigned to Reynolds Metals Co. [Cl. 53-17.] Stresses are relieved in thermoplastically sealed carton flaps by reheating and pressing the folded flaps.

Frattinger, Peter A. Bundle wrapping machine. U. S. patent 2,677,222. Filed June 28, 1950. 6 claims. Assigned to Robert Auguste Durand. [Cl. 53-45.] Bundles of large items such as sheets of plywood are wrapped with paper or the like applied as upper and lower covering sheets, then folded and fastened to make a complete package.

Hall, Winfield E., Jr., Hall, Robert E., and Perriguey, Forrest B. Container, apparatus and method for making the same. U. S. patent 2,677,489. Filed Aug. 9, 1949. 10 claims. Assigned to American Trust Co. [Cl. 229-4.5] Convolutely wound cylindrical container with fracture line formed at the top during winding of the container.

Heys, Ralph C. Slice mechanism for papermaking machines. U. S. patent 2,677,316. Filed June 19, 1950. 11 claims. Assigned to Millspaugh Ltd. [Cl. 92-44.] An adjustable nozzle, a leveling roll, and an inclined board as a final leveling means are contained in a channel whose lower side comprises an adjustable apron that is feathered where the stock is delivered to the wire.

Maynes, Harold H. Bag, composite material, and method of making. U. S. patent 2,677,496. Filed Sept. 22, 1950. 3 claims. Assigned to Arkell Safety Bag Co. [Cl. 229-55. Laminated bag material comprises crinkled kraft paper with glass fiber laminated in asphaltic material containing rubber or other elastomer.

Ohrn, Rudolph E. Sheet feeding mechanism. U. S. patent 2,677,543. Filed June 8, 1950. 2 claims. Assigned to Container Corporation of America. [Cl. 271-44.] Feed dogs on a rocking shaft engage the rearward edge of the lowermost sheet in a stack and move the sheet forward to be engaged by the main feed element.

Potdevin, Adolph. Apparatus for forming a valve in a bag and applying a supplemental sleeve forming sheet thereto. U. S. patent 2,677,319. Filed Sept. 23, 1950. 22 claims. Assigned to Potdevin Machine Co. [Cl. 93-8.] A sleeve sheet is inserted in a folded corner of bags having gusseted sides and the completed valve bag ejected from the machine.

Torudd, Erik, and Andersson, Nils. Method and machine for manufacturing container covers or lids of paper. U. S. patent 2,677,318. Filed Jan. 22, 1951. 6 claims. [Cl. 93-1.3.] Section cut from a laminated paper tube is fitted with disk closures cut from a supply roll to form cover for a cylindrical container.

Vogt, Clarence W. Method for making bundles of enwrapments. U. S. patent 2,677,317. Filed July 2, 1949. 4 claims. [Cl. 93-1.] Folded wrappings are bound in bundles by adhesive-coated tape with the folded edges of the wrappings held by the tape.

Wauda, George E. Interfitting lock for containers. U. S. patent 2,677,493. Filed March 14, 1949. 1 claim. Assigned to Marathon Corp. [Cl. 229-35.] Notched extensions of the end walls interlock with slits in the side walls.

### May 11, 1954

Borchardt, Lester F., and Felt, Clarence E. Bag closure. U. S. patent 2,678,154. Filed Sept. 29, 1948. 1 claim. Assigned to General Mills, Inc. [Cl. 229-62.] Mechanism and method of folding bag closures and sealing same.

Buttery, Kenneth T. Collapsible covered container or box. U. S. patent 2,678,153. Filed Feb. 6, 1952. 3 claims. Assigned to Sutherland Paper Co. [Cl. 229-36.] Notched tongues on the cover flap engage in inverted T-shaped notches in the front wall to provide a locking closure.

Coakley, Lige. Bag closing and sealing machine. U. S. patent 2,677,924. Filed Oct. 25, 1951. 13 claims. Assigned to Bemis Bro. Bag Co. [Cl. 53-148.] Top closures of large paper bags are folded, adhesive applied, and the folding completed with the projecting ears adhesively secured across the top of the bag.

Ens, Emil J. Method and apparatus for pleating blanks of laminated sheet material and preformed blanks of laminated sheet material. U. S. patent 2,677,993. Filed June 28, 1949. 6 claims. Assigned to Magnus Harmonica Corp. [Cl. 93-1.] Laminated blanks consisting of a relatively stiff paper ply and a flexible ply such as plastic film are fed into a pleating machine without interruption between blanks.

Goumeniouk, Gleb I. Stock inlet for papermaking machines. U. S. patent 2,677,991. Filed Feb. 2, 1948. 14 claims. Assigned to Powell River Co. Ltd. [Cl. 92-44.] Inverted U-shaped headbox comprises a stock inlet area with rectifier rolls, a still pond at the top under an air head, then an acceleration zone terminating in the slice over the forming wire.

Hornbostel, Lloyd. Stock distributor for papermaking machines. U. S. patent 2,677,992. Filed Feb. 5, 1949. 7 claims. Assigned to Beloit Iron Works. [Cl. 92-44.] Pair of conduits introduce stock into chambers on either side of a baffle plate, the open bottom of the chambers positioned above a rectifier roll that mixes stock from the chambers and eliminates eddy currents.

Ohlson, Paul E., and Hodge, Edward A. Steam-heated drier. U. S. patent 2,677,898. Filed Jan. 6, 1950. 18 claims. Assigned to The Pusey & Jones Corp. [Cl. 34-124.] Annular space is formed inside drier by light weight shell mounted within the drum. Steam is introduced into the annular space and condensate is directed to the shell interior for removal.

Ohlson, Paul E., and Hodge, Edward A. Jacketed steam drier. U. S. patent 2,677,899. Filed May 16, 1951. 11 claims. Assigned to The Pusey & Jones Corp. [Cl. 34-124.] Closely spaced ($^3/_8$-in.) concentric cylinders are divided into longitudinal compartments by condensate-collecting vanes with each compartment containing steam inlet and condensate-removal systems.

Schneider, David W. Method of and means for automatically picking waste material from carton blanks and like articles. U. S. patent 2,677,994. Filed Sept. 2, 1949. 16 claims. Assigned to Miehle Printing Press and Manufacturing Co. [Cl. 93-58.] Picking spurs are applied by a special device to engage or remove them from the stripping drum.

Shepherd, James C. Manifolding checkbook. U. S. patent 2,678,223. Filed Oct. 15, 1951. 4 claims. Assigned to Universal Business Forms, Inc. [Cl. 282-23.] Checkbook with detachable transfer means and record sheet facilitates account keeping.

Vander Lugt, Thomas, Jr. Collapsible carton member. U. S. patent 2,678,152. Filed Dec. 8, 1951. 2 claims. Assigned to Sutherland Paper Co. [Cl. 229-35.] Tray-type carton with double triangular corners folded against the outside of the box.

### May 18, 1954

Andriot, Lawrence J., Jr. Cordage package. U. S. patent 2,678,724. Filed March 30, 1953. 2 claims. Assigned to Puritan Cordage Mills. [Cl. 206-56.] Double package may be severed along a line of weakness to enable purchaser to buy a single or double hand of cordage.

Bonini, John H. Packaging device. U. S. patent 2,678,770. Filed Aug. 28, 1951. 2 claims. Assigned to Marathon Corp. [Cl. 229-87.] Two layers of sausage are enclosed with the upper layer retained by a band cut from the sleeve which encloses the lower layer.

David, Andrew M. Two-in-one bank deposit envelope. U. S. patent 2,678,769. Filed April 2, 1951. 1 claim. [Cl. 229-73.] Bank deposit slip and envelope may be reversed to provide for two mailings.

Eaton, Joseph K. Device for setting-up collapsible cardboard cartons. U. S. patent 2,678,590. Filed Nov. 25, 1949. 3 claims. Assigned to Bloomer Bros. Co. [Cl. 93-53.] Conical rollers initiate the erection as the folded blank is advanced on the conveyor and a shoe element completes the erection with a minimum of friction.

Ferguson, Kenneth C., and Schaefer, Norbert F. Shipping container. U. S. patent 2,678,765. Filed June 9, 1950. 1 claim. Assigned to Anderson Box Co. [Cl. 229-6.] Ventilated baby chick box of corrugated board with central partitions.

Fleming, William M., and Klein, Morris. Machine for manufacture of rectangular containers. U. S. patent 2,678,591. Filed June 16, 1950. 7 claims. Assigned to Eskimo Pie Corp. [Cl. 93-55.1.] Paper milk containers are assembled from a flattened tubular body blank and top and bottom closures adhesively secured into the erected body. Interior of the completed container may be waxed with a wax spray head.

Goss, Worth C. Method of pressing sheet lumber. U. S. reissue patent 23,839. Filed Feb. 6, 1953. 11 claims. Assigned to The Sheetwood Products Co. [Cl. 18-47.5.] Wet-ground brown rotted wood soluble in dilute alkali is claimed as the bonding agent in this reissue of U. S. patent 2,542,052 (Feb. 20, 1951).

Leighton, Philip S. Process of cast-coating paper. U. S. patent 2,678,890. Filed Jan. 22, 1951. 3 claims. Assigned to S. D. Warren Co. [Cl. 117-64.] Pool of water is held in the nip where the coated sheet is brought into contact with the casting drum. Water is supplied to and removed from this pool to prevent accumulation of coating composition.

Levkoff, David. Box or carton. U. S. patent 2,678,766. Filed Sept. 25, 1951. 3 claims. [Cl. 229-16.] Tray-type carton with hinged window cover and double end walls for packaging fruits and baked goods.

Schnuck, Carl F., and Johnson, Emil H. Calender and the like. U. S. patent 2,678,465. Filed Jan. 4, 1951. 5 claims. Assigned to Farrel-Birmingham Co., Inc. [Cl. 18-2.] Rolls are urged together by fluid pressure transmitted by hydraulic cylinders attached to the roll journals. Pressures are not subject to change because of expansion of parts while running.

Spencer, Virgil. Calender. U. S. patent 2,678,464. Filed June 13, 1950. 1 claim. Assigned to Armstrong Cork Co. [Cl. 18-2.] Short rod section with ball and socket joints is used between the adjusting screw and roll journal so that the bearings will not bind even under extreme adjustment.

Stone, William A. Stable liquid chlorine dioxide composition. U. S. patent 2,678,922. Filed Dec. 28, 1951. 4 claims. Assigned to Mathieson Chemical Corp. [Cl. 252-187.] Mixture of liquid chlorine and liquid chlorine dioxide in proportions by weight not exceeding one part of chlorine dioxide for each two parts of chlorine is claimed stable and free from danger of explosion.

Toensmeier, Patrick A. Carton for like articles. U. S. patent 2,678,767. Filed Oct. 24, 1950. 7 claims. Assigned to The Bartgis Brothers Co. [Cl. 229-40.] Sleeve-type carton to hold three cans by engagement of the chimes with inwardly folded flaps.

Vergobbi, Robert W. Reinforced bag, U. S. patent 2,678,768. Filed April 14, 1950. 1 claim. Assigned to Pneumatic Scale Corp., Ltd. [Cl. 229-55.] Reinforcing bottom strip of carton board or the like is inserted in a bag to make it self-supporting in an upright position.

Wait, Edward B. Dyeing fibrous sheet material. U. S. patent 2,678,588. Filed Oct. 28, 1948. 6 claims. Assigned to Masonite Corp. [Cl. 92-39.] Hardboard is dyed by spraying the top surface of the pad with dye solution, then pressing which causes part of the dye to migrate to the screen side for uniform color on both sides. Acid or direct dyes are suitable.

### May 25, 1954

Armstrong, Thomas J., and Iler, Ralph K. Process of preparing a hydrogen peroxide bleach. U. S. patent 2,679,483. Filed Oct. 26, 1951. 1 claim. Assigned to E. I. du Pont de Nemours & Co. [Cl. 252-186.] Sodium silicate uniformly effective in bleaching solutions is produced by mixing sodium silicate with sodium hydroxide solution to produce a ratio of silicon dioxide to sodium oxide from 2.25:1 to 3.75:1 and containing at least 5% silicon dioxide. The temperature rises above 60°C. and the mixture is aged at least five minutes before diluting and mixing with hydrogen peroxide.

Cram, Harvey G. Disk screen. U. S. patent 2,679,193. Filed Jan. 13, 1949. 4 claims. Assigned to Combined Locks Paper Co. [Cl. 92-34.] Casing is divided into two parts by a screen plate of slightly conical shape provided with runners on either side to exert a wiping action across the screen plate. Pulp is introduced on the concave side for improved efficiency.

Ehrenfried, John F., and Triche, Junius A., Jr. Thermosealable package. U. S. patent 2,679,194. Filed March 10, 1952. 7 claims. Assigned to The Goodyear Tire & Rubber Co. [Cl. 93-8.] Paper slip sheet is used to prevent consolidation of film where edge seal is used to form bags.

Mullinix, Charles D. Tear strip package and blank therefor. U. S. patent 2,679,349. Filed Sept. 30, 1948. 6 claims. [Cl. 229-40.) Sealed, perforated tear strip is incorporated in the blank structure for frozen food package.

Pomper, Anthony W., and Waechter, Charles J. Web coating apparatus. U. S. patent 2,679,231. Filed Sept. 7, 1951. 22 claims. Assigned to John Waldron Corp. [Cl. 118-63.] Air nozzle formed of metal plates adjustable for opening is mounted to remove excess coating from a paper web and is fitted with a baffle just below the nozzle to deflect coating particles and prevent build-up on the web.

Soffa, Albert. Carry bag handle construction. U. S. patent 2,679,350. Filed March 7, 1950. 2 claims. Assigned to Wolf Brothers, Inc. [Cl. 229-54.]

Soffa, Albert. Carry bag handle construction. U. S. patent 2,679,351. Filed March 7, 1950. 2 claims. Assigned to Wolf Brothers, Inc. [Cl. 229-54.]

Tichenor, Harold D. Chick box. U. S. patent 2,679,348. Filed Jan. 21, 1949. 2 claims. Assigned to Wabash Fibre Box Co. [Cl. 229-15.] Slotted cover slips over tabs in central partition to lock cover in place.

Whitton, Ernest C. Box scoring device. U. S. patent 2,679,-104. Filed July 17, 1953. 3 claims. [Cl. 33-42.] Adjustable hand tool for packers scores carton walls to make the carton fit the contents.

Whytlaw, Graeme G. Apparatus for forming tubular articles. U. S. patent 2,679,195. Filed July 13, 1950. 8 claims. Assigned to American Viscose Corp. [Cl. 93-82.] Web of paper or the like is formed into a tube with overlapped edges and a thread of thermosensitive material fed into the overlap, then heat and pressure applied to make the seal.

### June 1, 1954

Adler, Erich, and Hägglund, Erik K. M. Method of producing water-soluble products from black liquor lignin. U. S. patent 2,680,113. Filed Dec. 19, 1950. 19 claims. Assigned to Svenska Cellulosaforeningens Centrallaboratorium. [Cl. 260-124.] Lignin in alkaline black liquors is solubilized by treatment with an oxidizing gas, then a sulphonating agent and an aldehyde to produce a material valuable for tanning.

Becher, Hubert L. Fire-resistant product from comminuted woody material, urea, or melamine-formaldehyde, chlorinated hydrocarbon resin, and hydrated alumina. U. S. patent 2,-680, 102. Filed July 3, 1952. 7 claims. Assigned to Homasote Co. [Cl. 260-17.3.] Product may be molded as sheets or as formed items and are fire resistant as tested by the "Crib Test."

Brook, Leonard. Wrapping machine. U. S. patent 2,679,765. Filed April 21, 1952. 5 claims. Assigned to The Forgrove Machinery Co. Ltd. (Cl. 74-470.] Method of mounting a spring in the driving arm linkage for protection in case of jamming.

Davidson, John B., and Santelli, Thomas R. Method of sizing paper. U. S. patent 2,680,073. Filed Jan. 10, 1951. 6 claims. Assigned to Allied Chemical & Dye Corp. [Cl. 92-21.] Organosilane added to beaten pulp in conjunction with alum is claimed to produce a water-repellent paper.

Doyle, James F., and Onasch, Paul L. Method of making crinkled laminated material. U. S. patent 2,679,887. Filed July 22, 1949. 6 claims. Assigned to Arkell Safety Bag Co. [Cl. 154–33.05.] Paper coated or laminated with a film of polyethylene is crinkled by passing through a bath of water heated to condition paper and film, then doctored from a heated roll.

Dubin, Arthur. Display carton. U. S. patent 2,679,926. Filed Jan. 15, 1953. 2 claims. Assigned to Chemical Sundries Co. [Cl. 206–45.31.] Single blank is folded into a carton with an extension of one side wall forming a flap for display panel or directions.

Fridolph, John S. Delaminating composite web. U. S. patent 2,679,986. Filed June 19, 1950. 3 claims. Assigned to

Oxford Corp. [Cl. 242–66.] Plastic web and backing sheet of paper, fabric, or the like used in formation of the plastic are stripped apart and rewound separately under adjustable tension.

Frum, Herbert D. Foldable cardboard beach table. U. S. patent 2,680,047. Filed June 1, 1951. 3 claims. [Cl. 311-35.]

Goldberg, Arthur G. Automatic serving tray. U. S. patent 2,679,971. Filed March 22, 1952. 11 claims. Assigned to Standard Box Co. [Cl. 229-28.] Paperboard tray for foods and beverages.

Huebner, William C. Method for making sheet or web material. U. S. patent 2,680,079. Filed April 4, 1951. 1 claim. Assigned to The Huebner Co. [Cl. 117–93.] Electrostatic method and machine for the deposition of fibrous materials upon a wire or felt support by passing the ionized suspension through an electromagnetic field is claimed. Papermaking pulps at 60–70% consistency are used in the device.

Lockhart, Nathan J. Apparatus for separating airborne asbestos material. U. S. patent 2,679,933. Filed April 24, 1950. 5 claims. [Cl. 209-139.] Crusher for raw asbestos transports fiber by air blast from the crusher and classifies particles by arrangement of baffles within the separating chamber.

Madden, Wilson H. Frozen confection sandwich container. U. S. patent 2,680,557. Filed Feb. 9, 1950. 2 claims. [Cl. 229–38.]

Marrone, John C. Method of forming paper. U. S. patent 2,680,072. Filed Sept. 29, 1949. 1 claim. Assigned to A. M. Meincke & Sons, Inc. [Cl. 92–21.] Acid-treated cold swelling starch with calcium sulphate or other inert filler is claimed not to reduce the brightness of paper pulp.

Nielsen, Morris L., and Arnold, Russell W. Flameproofing composition of nitrogen-phosphorus compound in combination with organic compound having free hydroxy radicals. U. S. patent 2,680,077. Filed Dec. 30, 1949. 6 claims. Assigned to Monsanto Chemical Co. [Cl. 106-15.] Polyphosphorylamide and an alkyd resin are combined to form an intumescing coating composition.

Saidel, William R. Folding carton. U. S. patent 2,679,970. Filed June 17, 1949. 2 claims. [Cl. 229–27.] Tray-type carton with transverse partitions is formed from single sheet of paperboard.

Skardal, Karl A. Refiner for fiber suspensions and pulp. U. S. patent 2,679,788. Filed July 1, 1953. 4 claims. [Cl. 92–20.] Perforated, toboggan-shaped elements are carried by a rotating shaft within a perforated drum. Pulp introduced into the drum is dewatered between the elements and the drum wall and friction between same produces refining action.

Süs, Oskar. Light-sensitive diazotype material. U. S. patent 2,680,074. Filed Feb. 7, 1951. 7 claims. Assigned to Keuffel & Esser Co. [Cl. 95-6.]

Whitlock, William R. Cutterhead for debarking logs. U. S. patent 2,679,870. Filed Jan. 28, 1952. 4 claims. Assigned 33⅓% to Clark W. Adams. [Cl. 144-230.] Cutter head is flexibly mounted to compensate for bends in the log. Cutting teeth are easily replaceable and securely locked in the head.

### June 8, 1954

Barthel, Justus C., and Hastings, Randall. Tall oil rosin sizes stabilized with phenothiazine plus diarylamines. U. S. patent 2,680,694. Filed July 11, 1951. 6 claims. Assigned to American Cyanamid Co. [Cl. 106-218.] Tall oil rosin may be stabilized by addition of 0.5–2% of phenothiazine and about 0.2–2% of diarylamine based on weight of rosin.

Currie, Grover C. Carrier for cylindrical objects. U. S. patent 2,680,556. Filed Aug. 15, 1950. 1 claim. Assigned to Dacam Corp. [Cl. 229-37.] Can carrier.

Dearing, William C. Paper of improved wet strength. U. S. patent 2,680,682. Filed Jan. 26, 1950. 4 claims. Assigned to Allied Chemical & Dye Corp. [Cl. 92–3.] Paper made with a resin produced by reacting urea or melamine, formaldehyde, and a substituted carboxylic acid is claimed to possess superior wet-strength properties.

Dearing, William C. Paper treating resin. U. S. patent 2,680,734. Filed Jan. 26, 1950. 2 claims. Assigned to Allied Chemical & Dye Corp. [Cl. 260-70.] Condensation product of urea and formaldehyde, an alkali metal salt of glycolic acid, and ethylene glycol is claimed to impart greater wet strength to paper and to possess improved stability.

Grassie, Vernon R., and Wallis, Calvin R. Alkali cellulose preparation. U. S. patent 2,680,737. Filed May 17, 1951. 7 claims. Assigned to Hercules Powder Co. [Cl. 260-233.] Comminuted cellulose is slurried with aqueous sodium hydroxide containing isopropanol to produce a uniform alkali cellulose.

Laughlin, Kenneth C. Preparation of alkali cellulose. U. S. patent 2,680,738. Filed April 11, 1952. 6 claims. Assigned to Hercules Powder Co. [Cl. 260-233.] Very similar to U. S. patent 2,680,737. See above.

Lewis, Gilbert L. Laminating machine. U. S. patent 2,680,-468. Filed Aug. 1, 1949. 3 claims. [Cl. 154-1.] Laminating films are applied to one or both sides of a base sheet, then the laminate passed between a series of rolls individually adjustable as to temperature and nip pressure between each pair of rolls.

Mai, George W. Opening device for cartons. U. S. patent 2,680,558. Filed Jan. 10, 1951. 1 claim. Assigned to Gaylord Container Corp. [Cl. 229-51.] Sealing flap for ice cream carton is provided with line of perforations to confine tearing to a limited zone.

Meyers, George L. Laminated sheet material and method of making same. U. S. patent 2,680,700. Filed Sept. 26, 1950. 13 claims. Assigned to The Eagle-Picher Co. [Cl. 154-139.] Layers of formaldehyde-impregnated paper are bonded to a flexible elastomer base such as natural or synthetic rubber.

Obenshain, David N. Continuous digester chip level indicator. U. S. patent 2,680,298. Filed Dec. 6, 1951. 9 claims. Assigned to West Virginia Pulp & Paper Co. [Cl. 33-126.] Amount of movement of a periodically rotated arm is translated into an electrical impulse that controls a servomechanism which in turn controls the chip feeding device.

Obenshain, David N. Charging and discharging mechanism for use in continuous cooking of chips in the manufacture of pulp. U. S. patent 2,680,683. Filed Dec. 6, 1951. 11 claims. Assigned to West Virginia Pulp & Paper Co. [Cl. 92-7.] Disk valve for metering chips into or out of a digester comprises a rotating disk with chambers spaced around the perimeter and a metering device that prevents completely filling the pockets, thereby protecting the sliding surfaces from wear due to shearing chips. Sealing plates on either side of the disk contain ports through which the charge is received or emptied. Internal pressures are balanced by means of conduits from the digester.

Obenshain, David N. Strainer mechanism for use in continuous cooking of chips in the manufacture of pulp. U. S. patent 2,680,684. Filed Dec. 6, 1951. 8 claims. Assigned to West Virginia Pulp & Paper Co. [Cl. 92-7.] Concentric rings of overlapped plates allow removal of cooking liquors, and backwashing ot the strainer by segments prevents clogging.

Olson, Harry S., and Danison, Robert C. Transparent flame-resistant cellulose acetate butyrate. U. S. patent 2,680,691. Filed Dec. 16, 1948. 6 claims. Assigned to Diamond Alkali Co. [Cl. 106-15.] Composition suitable for injection or compression molding is made from cellulose acetate butyrate and a chlorinated paraffin wax with a stabilizer such as propylene oxide.

Poczatek, Frank G. Carbon paper for billing machines and the like. U. S. patent 2,680,629. Filed July 21, 1952. 3 claims. [Cl. 282-19.] Strip of carbon paper is fan-folded between holding sheets and secured with adhesive tabs.

Prince, Clarence F. Cracker wrapping machine. U. S. patent 2,680,336. Filed Dec. 28, 1950. 8 claims. Assigned to Package Machinery Co. [Cl. 53-131.] Stacks of fragile items are wrapped in heat-sealable material.

Rubin, Milton D. Method of manufacturing a conductive coated sheet and said sheet. U. S. patent 2,680,699. Field April 21, 1952. 12 claims. [Cl. 154-121.] Conductive metal-coated paper is laminated to phenolic resin base sheet.

Young, Lewis D. Display box. U. S. patent 2,680,512. Filed June 2, 1951. 1 claim. Assigned to Douglas Young, Inc. [Cl. 206-45.13.] Tray in bottom section is raised to inclined position upon opening cover.

Youngren, Milton D. Animated greeting card. U. S. patent 2,680,328. Filed March 17, 1952. 4 claims. Assigned to Hall Brothers, Inc. [Cl. 46-37.]

### June 15, 1954

Baker, Chester L., and Sams, Robert H. Water-resistant silicate adhesives for the machine fabrication of paperboard. U. S. patent 2,681,290. Filed Jan. 3, 1952. 20 claims. Assigned to Philadelphia Quartz Co. [Cl. 106-79.] Silicate adhesive containing 10% vegetable protein and 5% starch has a working life of 15 minutes to 8–12 hours.

Barney, Freeman, and Lee, James W. Paper cutter. U. S. patent 2,681,104. Filed Oct. 9, 1950. 2 claims. Assigned to The Challenge Machinery Co. [Cl. 164-54.] Clamping bar is actuated to apply clamping pressure on the stack a moment before and release pressure a moment after travel of the knife, thus guarding the knife at all times.

Bergstein, Robert M. Hollow wall box. U. S. patent 2,681,174. Filed Feb. 4, 1952. 3 claims. [Cl. 229-38.] Flaps formed by the window cut out in the double top are turned down to make the double wall construction of this collapsible box.

Brown, Charles K., Jr., and Smith, Gene C. Box loading and conveying machine. U. S. patent 2,681,171. Filed Nov. 30, 1951. 4 claims. Assigned to Robert E. Johnson. [Cl. 226-14.] Device loads filled containers such as paper milk bottles into a receiving carton by assembling groups of containers and lifting the groups into the carton.

Brown, Paul. Corrugated paper machine. U. S. patent 2,680,996. Filed Nov. 3, 1951. 12 claims. Assigned to Packaging Materials Corp. [Cl. 92-39.] Pulp slurry is flowed onto

the corrugated forming cylinder and vacuum applied to draw pulp into the grooves, then compressed by rolls which force water from the pulp. Molded sheet is dried and single or double backing sheets applied if intended for use in containers.

Christianson, Conrad J. Water reducible fire-retardant coating compositions. U. S. patent 2,681,326. Filed Aug. 26, 1950. 12 claims. Assigned to The Sherwin-Williams Co. [Cl. 260-39]. Amide-aldehyde resin with ammonium phosphate is water soluble and stable for several weeks and dries to a smooth film which intumesces when heated.

David, Andrew M. Double window envelope. U. S. patent 2,681,175. Filed June 17, 1952. 1 claim. [Cl. 229-75.] Return envelope which only requires refolding to expose the return address.

Ewing, Henry, and Blackmore, John S. Process and apparatus for making pile-surfaced material. U. S. patent 2,681,446. Filed Nov. 7, 1951. 4 claims. Assigned to British Celanese Ltd. [Cl. 117-17.] Fabric, paper, or the like is coated with adhesive, then with a floc of synthetic or natural fiber and the web passed through two high voltage fields to cause movement and orientation of the fibers on the coated surface. Any of several types of adhesive may be used that will set to a hard, flexible surface on the application of heat.

Ewing, Henry, and Black more, John S. Apparatus for distributing textile flock on a web. U. S. patent 2,681,036. Filed May 3, 1949. 4 claims. Assigned to Celanese Corporation of America. [Cl. 118-51.] Very similar to U. S. patent 2,681,446. See abstract above.

Fitchett, Thomas S. Creasing and slotting machine. U. S. patent 2,681,105. Filed May 6, 1949. 9 claims. [Cl. 164-60.] Creasing and slitting heads are supported on ways and the shafts are a driving means only. Rapidity of adjustment is claimed to be a feature of the device.

Frost, Orcutt W., and Tower, Gordon E. Method of making hardboard. U. S. patent 2,680,995. Filed Dec. 1, 1950. 16 claims. Assigned to Stimson Lumber Co. [Cl. 92-39.] Thermosetting resin and wax emulsion are added to fiber slurry under alkaline conditions and then precipitated on the fiber by addition of alum and acid before forming pad. Surface treatment of thermosetting resin and wax emulsion is applied to wet pad before hot pressing.

Guyer, Reynolds. Dome top carrier carton. U. S. patent 2,681,143. Filed Feb. 17, 1950. 7 claims. Assigned to Waldorf Paper Products Co. [Cl. 206-65.] Can carrier.

Guyer, Reynolds. Tray structure. U. S. patent 2,681,173. Filed Feb. 14, 1949. 4 claims. Assigned to Waldorf Paper Products Co. [Cl. 229-34.] Paperboard tray with double side wall construction for fruits and vegetables.

Huber, Dudley A. Apparatus for continuous chemical cleaning and conditioning of paper mill wet felts. U. S. patent 2,680,997. Filed May 8, 1953. 1 claim. [Cl 92-50.] Felt conditioning agent (Feltex) is sprayed under pressure of 40–60 p.s.i. to continuously condition wet felts.

McMillan, Archie, Beeker, Bernie, and Mickey, Arthur C Bellows folding machine. U. S. patent 2,680,998. Filed Jan. 24, 1950. 20 claims. Assigned to General Motors Corp. [Cl. 93-77.] Paper strip is formed into a tube and pleat formers on endless belts engage the tube to fold in accordion pleats.

Sanford, Roy S. Container. U. S. patent 2,681,172. Filed June 2, 1949. 10 claims. Assigned to The Autoyre Co., Inc. [Cl. 229-30.] Skeleton-structure tray is formed by cutting tongues from the bottom blank and folding upward to form side walls, a strip connecting the upper end of said tongues likewise holds the end walls in place.

Smart, Audrey C. Figure-forming foldable sheet. U. S. patent 2,680,935. Filed Sept. 21, 1951. 2 claims. [Cl. 46-124.] Decorated sheet is folded to form a child's bank.

Young, Lewis D. Display box. U. S. patent 2,681,141. Filed June 26, 1951. 7 claims. Assigned to Douglas Young, Inc. [Cl. 206-45.18.] Paperboard tray contains an insert with side and top of transparent material stapled to the false bottom of box claimed to be adapted to machine loading.

### June 22, 1954

Baxter, Joseph, Jr. Paper Machinery. U. S. patent 2,681,598. Filed June 4, 1951. 7 claims. Assigned to The Black-Clawson Co. [Cl. 92-26.] Dish-shaped impeller for tub refiner is made up of stepped annular rings coated with abrasive particles. Pulp passing across the face is forced to present new surfaces to the impeller as it progresses from face to face. Curved vanes are mounted in a radial manner on the face of the impeller to cause circulation within the tub.

Baxter, Joseph, Jr., Herrick, Wallace D., and Javid, Farhang. Wire stringing device for paper machinery. U. S. patent 2,681,721. Filed April 25, 1952. 12 claims. Assigned to The Black-Clawson Co. [Cl. 198-1.] Collapsible device supports the fourdrinier wire as it is unreeled, then projects supporting elements carrying the wire to deposit same on the machine.

Fridolph, John S. Paper coating machine. U. S. patent 2,681,636. Filed Jan. 15, 1951. 11 claims. Assigned to The Black-Clawson Co. [Cl. 118-249.] Coating rolls are mounted in bearing blocks on sliding wedges to permit accurate adjustment to predetermined settings. Coating composition is carried upward to an inverted fountain from which it is metered to the web.

Jacobs, Robert J. Paper embossing machinery. U. S. patent 2,681,611. Filed Jan. 26, 1951. 5 claims. Assigned to The Black-Clawson Co. [Cl. 101-23.] Lower roll of this embosser is urged upward by hydraulic cylinders located in the base. Bumpers of resilient material hold roll when pressure is released and keep drive gears meshed.

Kuenn, William W., and Jones, Elmer F. Method of packaging insulating strips. U. S. patent 2,681,702. Filed March 22, 1949. 7 claims. Assigned to Owens-Corning Fiberglas Corp. [Cl. 164-17.] Insulating batts are formed with the packaging sheet of perforated kraft paper adjacent the backing sheet; the batt is rolled and adhesively secured with gummed tape.

Palese, Richard A. M., and Young, Serenus H. A. Molding pulp and impregnating the product. U. S. patent 2,681,599. Filed Jan. 27, 1948. 4 claims. Assigned to American Cyanamid Co. [Cl. 92-55.] Molded pulp preforms are resin impregnated, the resin polymerized by oven curing and the preform subjected to a final molding process under heat and pressure to increase the density and attain a smooth surface.

Patton, Francis M., Jr. Method of preparing alkali cellulose. U. S. patent 2,681,908. Filed Sept. 25, 1950. 3 claims. Assigned to American Enka Corp. [Cl. 260-233.] Pulp sheets in the alkali soaking bath are supported at the upper corners until completely submerged by the caustic to insure more uniform treatment of pulp.

Prentice, Charles E. Log barking apparatus. U. S. patent 2,681,670. Filed May 21, 1951. 1 claim. [Cl. 144-208.] Rotating cutter head on arm is operated by motor mounted on carriage which travels on rails parallel to the long axis of the log.

Reimann, Kurt P. Means for embossing and printing. U. S. patent 2,681,612. Filed Jan. 31, 1951. 1 claim. [Cl. 101-25.] Flexible material is embossed and/or printed between a screen and an endless blanket as it passes around a heated drum equipped with pressure rollers to insure contact.

Rose, Alfred G., and Page, James. Suction feeding of sheets, cards, collapsed cartons, and the like. U. S. patent 2,681,803. Filed Sept. 6, 1950. 3 claims. Assigned to Rose Brothers (Gainsborough) Ltd. [Cl. 271-26.] Valve located near suction head is opened after head makes contact with sheet to make full suction force available as soon as valve is operated.

Schrock, Raymond L. Paper machinery. U. S. patent 2,681,610. Filed July 27, 1951. 5 claims. Assigned to The Black-Clawson Co. [Cl. 100-147.] Counterbalanced door construction for the reject ejection mechanism of a centrifugal pulp cleaner is claimed.

Simpson, George G. Coating apparatus for applying a resin in particulate form. U. S. patent 2,681,637. Filed Dec. 4, 1950. 2 claims. Assigned to Masonite Corp. [Cl. 118-308.] Resin particles are forced by a revolving brush through a screen or perforated plate to produce a uniform layer of particles later fused to a coating by application of heat.

Wallace, Lewis C. Method and apparatus for hydrolyzing cellulosic materials. U. S. patent 2,681,871. Filed Feb. 16, 1951. 16 claims. Assigned 30% to Sam M. Nickey, Jr., 30% to to W. Jeter Eason, 15% to Lawrence D. Kellogg, and 25% to United States Steel Corp. [Cl. 127-1.] Sawdust, wood chips, or the like are introduced at the top of the apparatus and dilute acid and steam at the bottom to continuously produce a product suitable for fermentation to ethanol or butanol or concentration to molasses for stock feed.

Way, Harvey H. Roll wrapping apparatus. U. S. patent 2,681,534. Filed April 23, 1951. 37 claims. Assigned to Powell River Co. Ltd. [Cl. 53-98.] Roll of paper is rotated to wind wrapper, ends of wrapper crimped flat against end of roll and end cap applied to complete wrapping.

Welshenbach, Charles D., and Gagen, Thomas H. Article positioning and cushioning device for use in shipping containers. U. S. patent 2,681,733. Filed Nov. 10, 1950. 5 claims. Assigned to The Hinde & Dauch Paper Co. [Cl. 206-62.] Strip of corrugated board is cut and folded to form inverted V's and locked with a tongue cut from the center of the strip.

Bivans, Elbert L. Boxmaking machine. U. S. patent 2,682,209. Filed Jan. 23, 1950. 14 claims. Assigned to E. L. Bivans, Inc. [Cl. 93-49.] Flattened boxes are erected and the bottom closure flaps tucked in place.

### June 29, 1954

Bivans, Elbert L. Boxmaking machine. U. S. patent 2,682,210. Filed Feb. 5, 1951. 13 claims. Assigned to E. L. Bivans, Inc. [Cl. 93-49.] Improvement on U. S. patent 92,82,209 (June 29, 1954).

Davis, Albert S., Jr. Wet bottom precipitator. U. S. patent 2,682,314. Filed Oct. 30, 1952. 3 claims. Assigned to Research Corp. [Cl. 183-7.] Sump of weak liquor is constantly agitated to dissolve solids from the electrodes and fortified liquors withdrawn and replaced by weak liquor.

Herrlinger, Paul W. Tape testing device. U. S. patent 2,682,171. Filed Aug. 9, 1952. 7 claims. Assigned to The Gummed Products Co. [Cl. 73-102.] Stay tape joints of boxboard cartons are tested in this device which clamps the corner between plates, then applies force to move the plates apart, rupturing the tape.

Larson, Marvin, Stedman, Allan L., and Dannemiller, Franklin T. Tote box. U. S. patent 2,682,362. Filed Feb. 6, 1951. 8 claims. Assigned to Convoy, Inc. [Cl. 229-23.] Open top box of strong construction with sloping side and end walls for ease in stacking.

Mitchell, Reid L. Aging alkali cellulose. U. S. patent 2,682,536. Filed May 22, 1952. 1 claim. Assigned to Rayonier Inc. [Cl. 260-233.] Cobalt salts are added to control aging of viscose.

Monroe, Charles Z., and Raymond, Norton G. Carton converting machine. U. S. patent 2,682,208. Filed April 15, 1948. 20 claims. Assigned to Ex-Cell-O Corp. [Cl. 93-36.] Paperboard is printed, scored, cut into blanks, and stripped of waste before delivery from the machine.

Rynish, Louis J. Machine for making and assembling spools. U. S. patent 2,682,207. Filed Sept. 7, 1951. 9 claims. Assigned to Central Spool and Tube Co. [Cl. 93-1.] Cores and end disks for ribbon spools are assembled in this machine.

Sax, Max. Method of making packages. U. S. patent 2,682,138. Filed March 22, 1952. 6 claims. Assigned to Container Mfg. Co. [Cl. 53-11.] Laminated cardboard sheets with registered holes form pockets to hold pills or the like in individual compartments sealed in back by foil membrane and in front by transparent film.

Vincent, Bertram J. Screen plate for papermaking machines. U. S. patent 2,682,205. Filed June 28, 1949. 5 claims. [Cl. 92-31.] Cut screen plates of stainless steel are fashioned without sharp angles to avoid cracking of the plates. Stiffening ribs in the plates likewise contribute to the strength of the plate.

Vogt, Clarence W. Apparatus for and method of chaining enwrapments. U. S. patent 2,682,206. Filed Oct. 28, 1948. 5 claims. [Cl. 93-1.] Bags are brought into overlapped relation and sealed together with thermoplastic adhesive for passage through the filling machinery, then the units separated.

# RECENT BOOKS

**Encyclopedia of Chemical Technology.** Vol. 11. Edited by Raymond E. Kirk and Donald F. Othmer, Polytechnic Institute of Brooklyn. The Interscience Encyclopedia, Inc., New York, 1953. Cloth, $7^1/_2 \times 10^1/_2$, 950 pages. Illustrated. $30.

**Encyclopedia of Chemical Technology.** Vol. 12. Edited by Raymond E. Kirk and Donald F. Othmer, Polytechnic Institute of Brooklyn. The Interscience Encyclopedia, Inc., New York, 1954. Cloth, $7^1/_2 \times 10^1/_2$, 955 pages. Illustrated. $30.

The present volumes are the eleventh and twelfth in a series of fourteen. Although the books have been issued at intervals of six months, the reviewer is pleased to note that the series is approaching completion. Another series of outstanding volumes, a set of five, reached the fourth volume at the time of the editor's death. The final volume of that set is in preparation by a group of individuals and will appear at least three years after the fourth volume.

The price of $30 refers to separate issues. For individuals and companies that have subscribed to the entire series, the price of each volume is $25.

Volume 11 covers the technology of subjects arranged alphabetically from Polyols to Rutin. Included in this range are such subjects as polysaccharides, potassium, potentiometry, powder metallurgy, power generation, pressure measurement, compressors, printing and reproducing, printing ink, protein fibers and plastics, pulp (prepared by R. S. Hatch), quality control, radicals, radiography, rayon and acetate, refractories, refrigeration, resins, rheology, rosin and derivatives, and rubber.

Volume 12 covers the technology of subjects treated alphabetically from Sabadine to Stilbestrol. Included in this range are such subjects as safety, salt, sampling, seaweed colloids, sedimentation, sewage, silicates and silicones, size measurement of particles, size reduction and separation, smokes and fumes, soap, sodium compounds, solvent recovery, soybeans, sprays, stains, starch, steam, and stereochemistry.

**Woodpulp.** By Erland O. Bjoorn, translated from the original Swedish text by J. P. Firrell and I. R. de Mare (England). Published by A/B Svensk Travaru-Tidnuig, Kungsgatan 17, Stockholm C., Sweden, 1954. Fabrikoid, $5 \times 7^1/_2$, 360 pages. $5.00.

A handbook relating to pulp and paper written principally for the benefit of executives on the buying and selling sides of the pulp and paper trades, especially in the Scandinavian countries. Mr. Bjoorn is now active as a wood pulp agent in Basle, Switzerland.

It deals with both technical and commercial aspects of the trade. Among the subjects covered are the following: Historical Survey; Papermaking Fibers giving brief descriptions of the various kinds of pulp together with a number of photomicrographs; Handling of Pulpwood; Manufacture of Woodpulp, by grades; Chemical By-products such as cymene, tall oil, alcohol, and turpentine; Testing Methods based on the standards of TAPPI, the Swedish, and German industries; Manufacture of Paper and Cellulose Derivatives; Sales Organizations and Methods, including contract forms; and Transportation, including chartering, transit insurance, and invoicing.

**Chemical Business Handbook.** Edited by John H. Perry, E. I. du Pont de Nemours & Co., Inc. McGraw-Hill Book Co., New York, 1954. Cloth, $7^1/_4 \times 10$, 1370 pages. $17.

A book prepared in 20 sections by 124 contributors in the McGraw-Hill Chemical Engineering Series. It reflects the trend throughout industry of training technically trained men and junior executives for advancement in management through knowledge of a wide range of subjects.

The subjects covered include: business finance, management and control by cost accounting and planning, commercial chemical development, research, market research, sources of information, industrial purchasing, production, traffic and transportation, sales, industrial advertising, credits and collections, personnel management, public relations, business law, patents and patent law, industrial toxicology, insurance and loss prevention, reports and report writing, and business mathematics.

Although the book is prepared for the chemical industries the pulp and paper industry to a certain extent is considered to be in this category. For the young men in the industry this book should be of considerable value.

*Note:* Books reviewed in this column may be obtained from the Technical Association of the Pulp and Paper Industry, 155 E. 44th St., New York 17, N. Y.

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# INDEX TO ADVERTISERS


American Cyanamid Co. (Paper Chemicals Dept.) ..... 97 A
American Cyanamid Co. (Dyestuffs Dept.) ..... 2nd Cover
Ansul Chemical Co. ..... 56 A
Appleton Machine Co. ..... 49 A
Appleton Wire Works ..... 29 A
Atlantic Refining Co. (Paraffin Waxes) ..... 23 A

Babcock & Wilcox Co. ..... 99 A
Baltimore & Ohio Railroad (Freight Dept.) ..... 13 A
Bauer Bros. Co. ..... 53 A
Beloit Iron Works ..... 1 A
Bird Machine Co. ..... 105 A
Bolton & Sons, Inc., John W. ..... 31 A
Brookfield Engineering Laboratories, Inc. ..... 196 A
Brown Co. ..... 44 A

Cabble Wire Company, Inc. ..... 123 A
Cameron Machine Co. ..... 109 A
Carpenter Steel Co. (Alloy Tube Div.) ..... 58 A
Chemipulp Process, Inc. ..... 40 A
Cities Service Oil Co. ..... 35 A
Columbia-Southern Chemical Corp. ..... 4th Cover
Corn Products Sales Co. ..... 113 A

Dicalite Division (Great Lakes Carbon Corp.) ..... 96 A
Dorr Co. ..... 98 A
Downingtown Manufacturing Co. ..... 19 A
du Pont de Nemours & Co., Inc., E. I. (Dyestuffs) ..... 2 A
du Pont de Nemours & Co., Inc., E. I. (Electrochemicals) ..... 29 A
du Pont de Nemours & Co., Inc., E. I. (White Pigments) ..... 107 A

Ebasco Services, Inc. ..... 128 A
Egan & Co., Frank W. ..... 41 A

Foxboro Co. ..... 40 A
Fuller Co., The ..... 94 A

Geigy Dyestuffs Div. (Geigy Chemical Corp.) ..... 129 A
General Chemical Div. (Allied Chem. & Dye Corp.) ..... 91 A
General Dyestuff Company ..... 102 A
Glidden Co., The ..... 101 A
Goslin-Birmingham Mfg. Co. ..... 57 A

Harris-Seybold Co. ..... 77 A
Heppenstall Co. ..... 21 A
Hercules Powder Company ..... 50 A–51 A
Hooker Electrochemical Co. ..... 33 A
Huber Corp., J. M. ..... 4 A
Huyck & Sons, F. C. ..... 48 A

Improved Machinery, Inc. ..... 65 A

Jackson & Church Co. ..... 79 A
Johns-Manville Corp. (Celite) ..... 52 A
Johns-Manville Corp. (Packings) ..... 9 A
Jones & Sons Co., E. D. ..... 59 A

Kamyr, Inc. ..... 62 A–63 A
Kelco Co., The ..... 7 A

Lawrence Pumps, Inc. ..... 60 A
Lobdell United Corp. ..... 119 A

Magnus Chemical Co. ..... 206 A
Mallinckrodt Chemical Works ..... 196 A
Mason-Neilan Regulator Co. ..... 42 A–43 A
Mixing Equipment Co., Inc. ..... 47 A
Monsanto Chemical Co. (Organic Div.) ..... 75 A
Morden Machines, Inc. ..... 83 A
Mt. Vernon-Woodberry Mills (Turner-Halsey Co.) ..... 71 A

National Aluminate Corp. ..... 81 A
National Aniline Div. (Allied Chem. & Dye Corp.) ..... 67 A
Newport News Shipbuilding & Dry Dock Co. ..... 121 A
Nopco Chemical Co. ..... 61 A

Pandia, Inc. ..... 100 A
Parsons & Whittemore, Inc. ..... 117 A
Penick & Ford, Ltd., Inc. ..... 125 A
Perkins & Son, Inc., B. F. ..... 92 A
Permutit Co., The ..... 89 A
Pusey & Jones Corp. ..... 37 A

Raybestos-Manhattan Inc. (Manhattan Rubber Div.) ..... 3 A
Reichhold Chemicals, Inc. ..... 85 A
Reliance Electric & Engineering Co. ..... 10 A–11 A
Rhodia, Inc. (Alamask Div.) ..... 127 A
Rice Barton Corp. ..... 73 A
Rohm & Haas Co. ..... 25 A
Roots-Connersville Blower (Div. of Dresser Industries, Inc.) ..... 45 A
Ross Engineering Corp., J. O. ..... 27 A
Ross Midwest-Fulton Corp. ..... 129 A

S & S Corrugated Paper Machinery Co., Inc. ..... 64 A
Sandusky Foundry & Machine Co. ..... 115 A
Sandy Hill Iron & Brass Works ..... 92 A
Smith Corp., A. O. ..... 95 A
Solvay Process Div. (Allied Chem. & Dye Corp.) ..... 46 A
Sprout, Waldron & Co., Inc. ..... 69 A
Standard Oil Co. (Indiana) ..... 111 A
Stebbins Engineering & Mfg. Co. ..... 12 A
Sutherland Refiner Corp. ..... 130 A–131 A–132 A–133 A

Taylor Instrument Companies ..... 8 A
Texas Gulf Sulphur Co. ..... 6 A
Thwing-Albert Instrument Co. ..... 40 A
Titanium Pigment Corp. ..... 3rd Cover

Valley Iron Works ..... 66 A
Vanderbilt, Co., R. T. ..... 15 A
Virginia Smelting Co. ..... 17 A

Waldron Corp., John ..... 87 A
Wallace & Tiernan, Inc. ..... 93 A
Westinghouse Electric Corp. ..... 54 A–55 A
Weyerhaeuser Timber Co. (Pulp Div.) ..... 68 A
Witco Chemical Co. ..... 92 A


## PROFESSIONAL SERVICES


The H. K. Ferguson Company ..... 205 A
G. D. Jenssen Co., Inc. ..... 205 A
Alvin H. Johnson & Co., Inc. ..... 205 A
Knowles Associates ..... 205 A
Chas. T. Main, Inc. ..... 205 A
Roderick O'Donoghue & Company ..... 205 A
Paper & Pulp Testing Laboratories ..... 205 A
Pfeifer & Shultz ..... 205 A
Reynolds, Smith & Hills ..... 205 A
Sandwell & Company ..... 205 A
Seelye Stevenson Value & Knecht ..... 205 A
J. E. Sirrine Co. ..... 205 A

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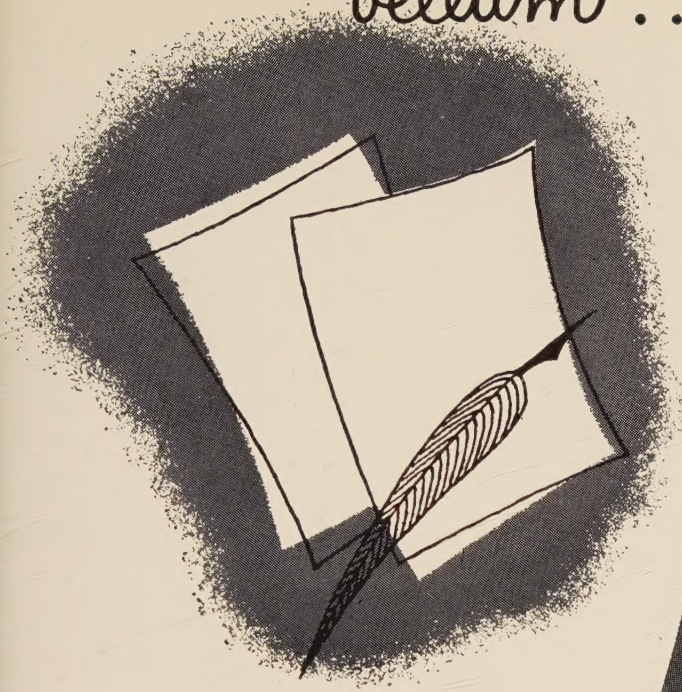

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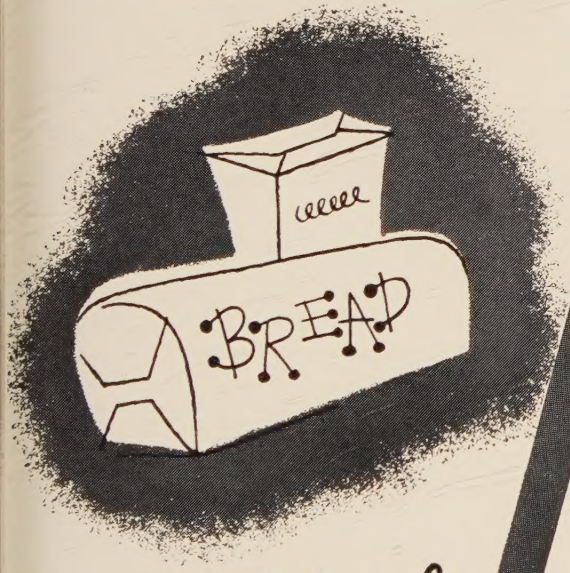


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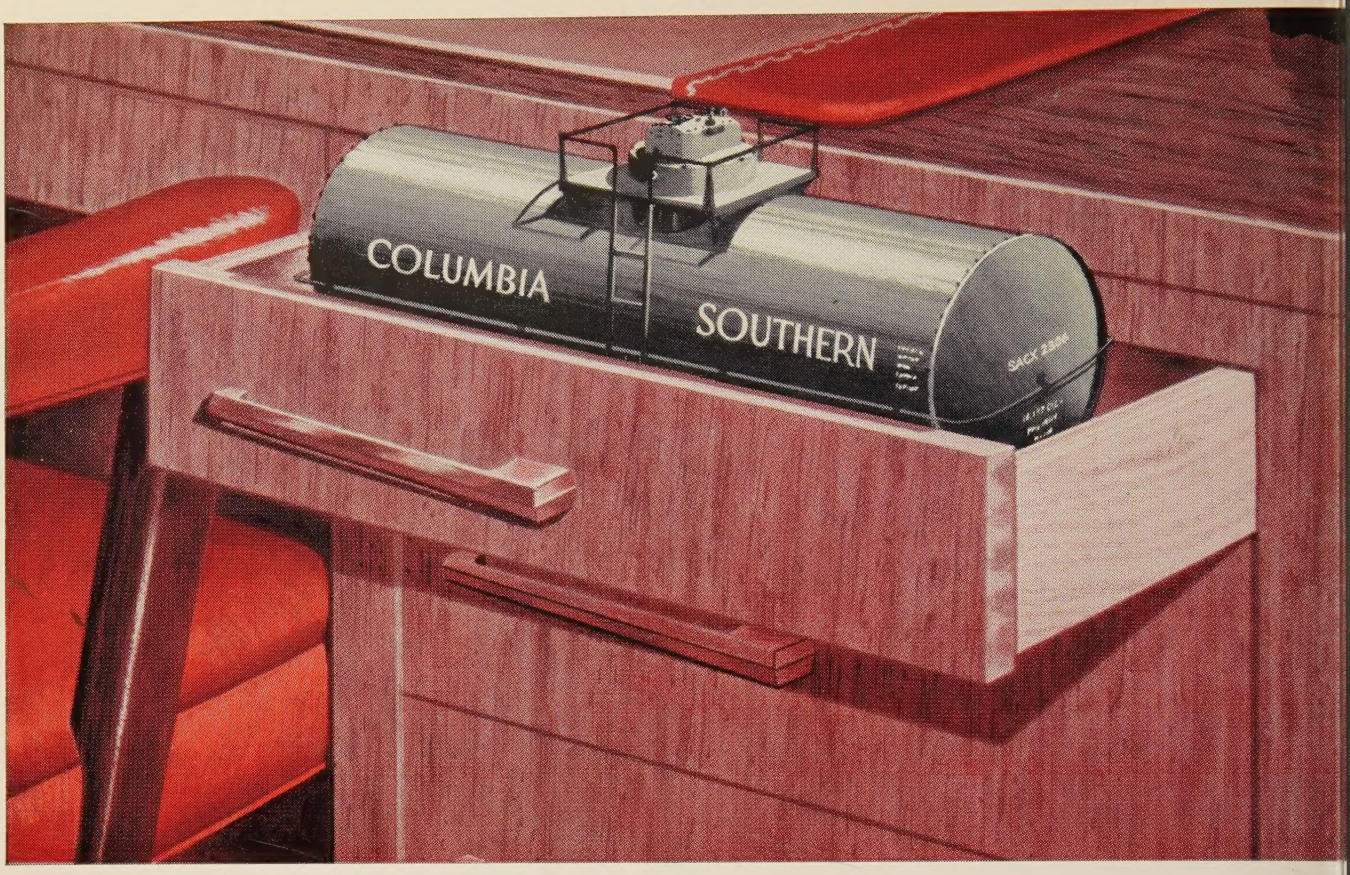


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